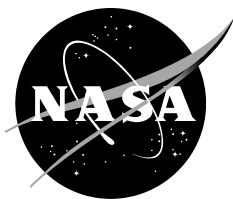


NASA/TM—2017–43; 7; 3



Method for the Collection, Gravimetric and Chemical Analysis of Nonvolatile Residue (NVR) on Surfaces

*Keith Gordon, Gugu Rutherford, and Denisse Aranda
Langley Research Center, Hampton, Virginia*

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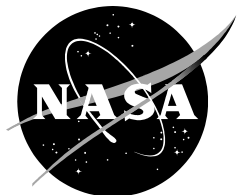
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*Keith Gordon, Gugu Rutherford, and Denisse Aranda
Langley Research Center, Hampton, Virginia*

National Aeronautics and
Space Administration

*Langley Research Center
Hampton, VA 23681*

Acknowledgments

We thank Mr. Matt Shaka from NASA Goddard Space Flight Center for all the training and valuable assistance provided in the development and implementation of a procedure for the collection, gravimetric and chemical analysis of Nonvolatile Residue (NVR) on surfaces. We would also like to thank Mr. Kenneth Smith (Langley Aerospace Research Student Scholars LARSS student) for the development of the Gas Chromatography and Mass Spectrometry (GCMS) software operations document. We would also like to thank Ms. Amanda Stark for the development of the Fourier Transform Infrared Spectroscopy (FTIR) operations for the cryogenic rinse document. We would also like to acknowledge Mrs. Patti Davis (NASA retiree/trainer) and Mr. Robert Edahl for assistance in performing the GCMS analysis, the sample preparation, and gravimetric residue determination for this report. We would also like to thank Dr. Robert Bryant for valuable discussions and assistance.

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Method for the Collection, Gravimetric and Chemical Analysis of Nonvolatile Residue (NVR) on Surfaces

Abstract

Nonvolatile residue (NVR), sometimes referred to as molecular contamination is the term used for the total composition of the inorganic and high boiling point organic components in particulates and molecular films deposited on critical surfaces surrounding space structures, with the particulate and NVR contamination originating primarily from pre-launch operations. The “nonvolatile” suggestion from the terminology NVR implies that the collected residue will not experience much loss under ambient conditions. NVR has been shown to have a dramatic impact on the ability to perform optical measurements from platforms based in space. Such contaminants can be detected early by the controlled application of various detection techniques and contamination analyses. Contamination analyses are the techniques used to determine if materials, components, and subsystems can be expected to meet the performance requirements of a system. Of particular concern is the quantity of NVR contaminants that might be deposited on critical payload surfaces from these sources. Subsequent chemical analysis of the contaminant samples by infrared spectroscopy and gas chromatography mass spectrometry identifies the components, gives semi-quantitative estimates of contaminant thickness, indicates possible sources of the NVR, and provides guidance for effective cleanup procedures. In this report, a method for the collection and determination of the mass of NVR was generated by the authors at NASA Langley Research Center. This report describes the method developed and implemented for collecting NVR contaminants, and procedures for gravimetric and chemical analysis of the residue obtained. The result of this NVR analysis collaboration will help pave the way for Langley’s ability to certify flight hardware outgassing requirements in support of flight projects such as Stratospheric Aerosol and Gas Experiment III (SAGE III), Clouds and the Earth’s Radiant Energy System (CERES), Materials International Space Station Experiment – X (MISSE-X), and Doppler Aerosol Wind Lidar (DAWN).

Introduction

Nonvolatile residue (NVR) resulting from the outgassing of materials used on spacecraft can cause electronic problems, serious attenuation of various optical signals, thermal control changes, and adhesion problems.¹⁻⁴ Mission success is dependent on the effects of NVR on critical spacecraft hardware. Failures in optical, thermal, and mechanical systems impact the life of a spacecraft and can result in the loss of millions of dollars.^{3,4} One approach to mitigate the risk from outgassed contamination, or NVR, is to choose low outgassing materials. The ASTM E595-84 standard method of determining total mass loss (TML) and collected volatile condensable materials (CVCM) is widely used to screen and validate candidate materials.^{4,5} The commissioning of materials solely by this standard does not ensure compliance with spacecraft cleanliness and performance requirements. However, ASTM E595-84 testing has become standard practice for assessing NVR risk. Nonetheless, good quality control to verify the space worthiness of materials before use in space systems is of extreme importance.

An alternative method of contamination control for space applications is to test components and subsystems in thermal vacuum chambers which provide a space-like environment for collecting NVR on cryogenically cooled surfaces.⁶ The NVR is collected on cold fingers and/or scavenger plates according to the method described in ASTM E834-81.⁷ A mass determination method is used to quantitatively determine the amount of NVR and Fourier Transform Infrared (FTIR) spectroscopy and/or Gas Chromatography Mass Spectrometry (GCMS) is used for chemical identification of the residue.¹⁻³ Understanding the chemistry of material outgassing or NVR helps to assist contamination control engineers in making technically sound risk assessments.

In this regard, the method used for the nonvolatile residue analysis performed by the contamination control team is described in detail. Step-by-step procedures for a nonvolatile residue analysis process are provided in the main body of the document. Subsequent appendix sections include analytical instrumentation software procedures and instructions developed, adapted and followed for the respective NVR activity. Adhering to these procedures without deviation enables the collection and determination of the mass and chemical composition of NVR samples.

Description of Method

Nonvolatile Residue NVR Collection and Detection

Nonvolatile residue collected in scavenger plates and cold fingers after the bake-out of a thermal vacuum chamber located in the Systems Integration & Test Branch SITB located at NASA Langley Research Center (NASA LARC) is extracted with 75 ml of isopropyl alcohol according to ASTM E834-81.⁷ The vast majority of NVR samples are collected from rinses off scavenger plates. Scavenger plates and cold fingers are both composed of 316 stainless steel (See Figures 1 and 2).^{2,7} These scavenger plates are cryogenically controlled which enables the plate to collect particulate from outgassing events while performing integration testing. A cold finger is a device that is used in a thermal vacuum chamber for obtaining residual condensable gas and particulates during integration testing, and during testing operations, the cold finger is the last cryogenically controlled surface to return to ambient temperature.⁶ Both the scavenger plate and the cold finger operate as a cohesive system for monitoring the NVR deposition onto flight hardware. The comparison of NVR deposited on the scavenger plate and the cold finger enables the analyst to create a detailed investigation of the chemical composition and the amount of NVR deposited on space hardware.



Figure 1. Scavenger Plate in Thermal Vacuum Chamber

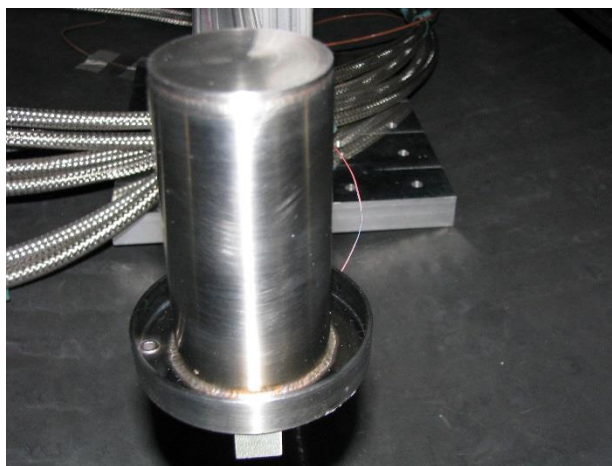


Figure 2. Cold Finger in Thermal Vacuum Chamber

Gravimetric Analysis: Mass Determination

Mass determination allows for a gravimetric measurement of the molecular residue that accumulates on cold finger(s) and scavenger plate(s). The ability to determine this contamination gravimetrically allows for a quantification of the effect of NVR on critical spacecraft components. During the manual evaporation method, the mass of the molecular residue obtained from the NVR rinse is measured on the pre-weighed aluminum weighing pan.^{7,8} In addition to this, a blank control sample, using the same solvent, is collected from a witness plate of similar dimensions as the NVR rinse witness plate. The mass is recorded for the blank control sample, the aluminum pan plus residue, and the clean aluminum pan. The mass measurements obtained should be within +/- 0.01mg for accurate and precise quantitative measurements. The calculations below are performed and a mass determination is obtained according to standards, ASTM E1235-12 and IEST-STD-CC1246E.⁸⁻¹⁰

$$\text{NVR} = (M_f - M_i) - M_b / V$$

M_f = mass of the aluminum pan plus residue, mg

M_i = mass of the aluminum pan, clean, mg

M_b = mass of blank NVR, mg

V = volumetric amount of NVR rinse sample added to aluminum pan, ml

This mass determination method requires the ability to work on a multi-dimensional surface since particles and molecular films can deposit on the front and backside of the witness plate. The environmental conditions for the testing are in cryogenic and high vacuum surroundings. The method outlined in the aforementioned ASTM standards allow for a more accurate account of the mass collected on the witness plate by using gravimetric and volumetric data to obtain a quantitative account of the NVR.^{7,8} Both standards will account for the environmental and surface variations. From the NVR calculation, the following table can be used to determine the NVR cleanliness levels. Determining a mass and identifying the NVR cleanliness level will allow for a complete and selective quantitative measurement of the cold finger(s) and scavenger plate(s).

Level ($\mu\text{g}/\text{cm}^2$ or $\mu\text{g}/0.1\text{cm}^3$)	Former Designation Level	Maximum allowable NVR limit mass / 0.1 m^2 or mass / 0.1 L
R1E-5	AA5	10 ng
R2E-5	AA4.7	20 ng
R5E-5	AA4.3	50 ng
R1E-4	AA4	100 ng
R2E-4	AA3.7	200 ng
R5E-4	AA3.3	500 ng
R1E-3	AA3	1 μg
R2E-3	AA2.7	2 μg
R5E-3	AA2.3	5 μg
R1E-2	A / 100	10 μg
R2E-2	A / 50	20 μg
R5E-2	A / 20	50 μg
R1E-1	A / 10	100 μg
R2E-1	A / 5	200 μg
R5E-1	A / 2	500 μg
R1	A	1 mg
R2	B	2 mg
R3	C	3 mg
R4	D	4 mg
R5	E	5 mg
R7	F	7 mg
R10	G	10 mg
R15	H	15 mg
R25	J	25 mg

Table 1. Nonvolatile residue cleanliness levels (IEST- STD-CC1246E)¹⁰⁻¹²

Chemical analysis

FTIR and GCMS (See Figure 3 and 4) are two powerful analytical techniques that can be used for examining the chemistry of material outgassing or NVR. FTIR spectroscopy is a measurement technique that allows one to record infrared spectra. Fundamentally, FTIR spectra illustrate absorption bands with characteristic frequency attributed to different functional groups within the molecule, such as carbonyls (C=O), alkyls (C-H), or multiple-bonded carbon atoms (C=C) that vibrate at characteristic frequencies due to various bending or stretching motions. The frequencies are in the infrared range from approximately $1,000$ to $3,500\text{ cm}^{-1}$ (10 to $2.9\text{ }\mu\text{m}$) and can be shifted by intramolecular and non-bonded, intermolecular interactions.^{13,14} Infrared light is guided through an interferometer and then through the sample. The sample preparation procedure for NVR is relatively simple, straight forward and fast and is called “casting a film.” This procedure involves dissolving the respective NVR in a small amount of solvent, transfer to a ZnSe (or the Attenuated Total Reflectance ATR crystal of choice), and evaporation of the solvent which leaves only the residual NVR film. FTIR has excellent sensitivity with the limit of detectability being

near 1 microgram. NVR typically consists of mixtures of compounds and FTIR analysis provides a readout of the chemical species present.

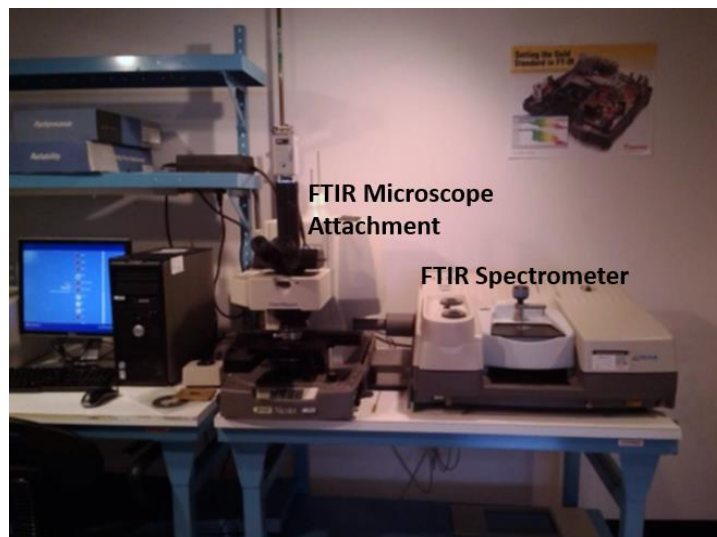


Figure 3. Infrared Spectroscopy Laboratory (Building 1293C room 153C).

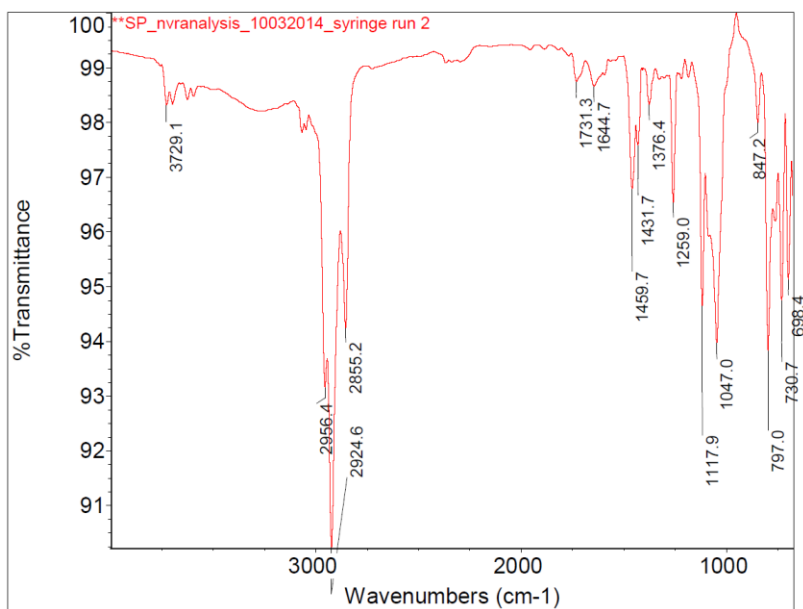


Figure 4. FTIR Spectrum of NVR from a Scavenger Plate.

Infrared analysis is more powerful and beneficial when coupled with other types of analysis. For example, GCMS is an analytical tool that is often used in conjunction with infrared spectroscopy in the characterization and structural determination of samples (Figures 3, 4 and 5). A GCMS is simply a gas chromatograph with a very large and very expensive detector, but one that can give a definitive identification of mixtures of compounds (See Figures 5 and 6).^{15,16} GCMS reveals the compounds eluted at different retention times with mass spectra corresponding to compounds present in a sample. The results of GCMS analysis are more revealing than FTIR and provides additional insights into the chemical nature of the NVR, including probable sources and possible consequences of the presence of these contaminants. Additionally, GCMS analysis may provide separation of complex mixtures, exact molecular identification of components, and profiles of the volatility characteristics of the NVR.¹ The sensitivity of GCMS analysis is at least an order of magnitude better than FTIR. Additional running of blank control samples and reference standards ensures the credibility and reliability of results.

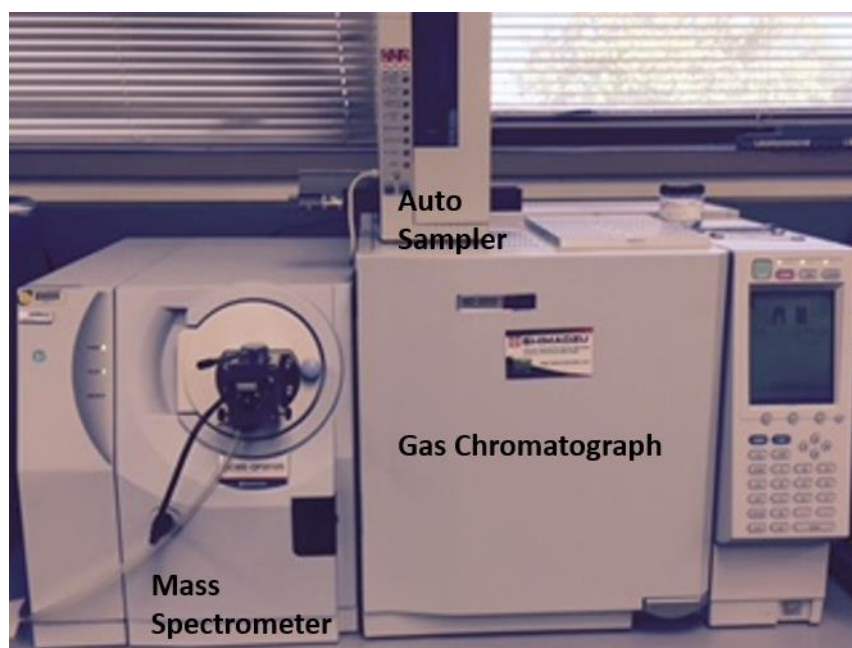


Figure 5. GCMS Chromatograph Equipment Setup (Building 1293C room 266).

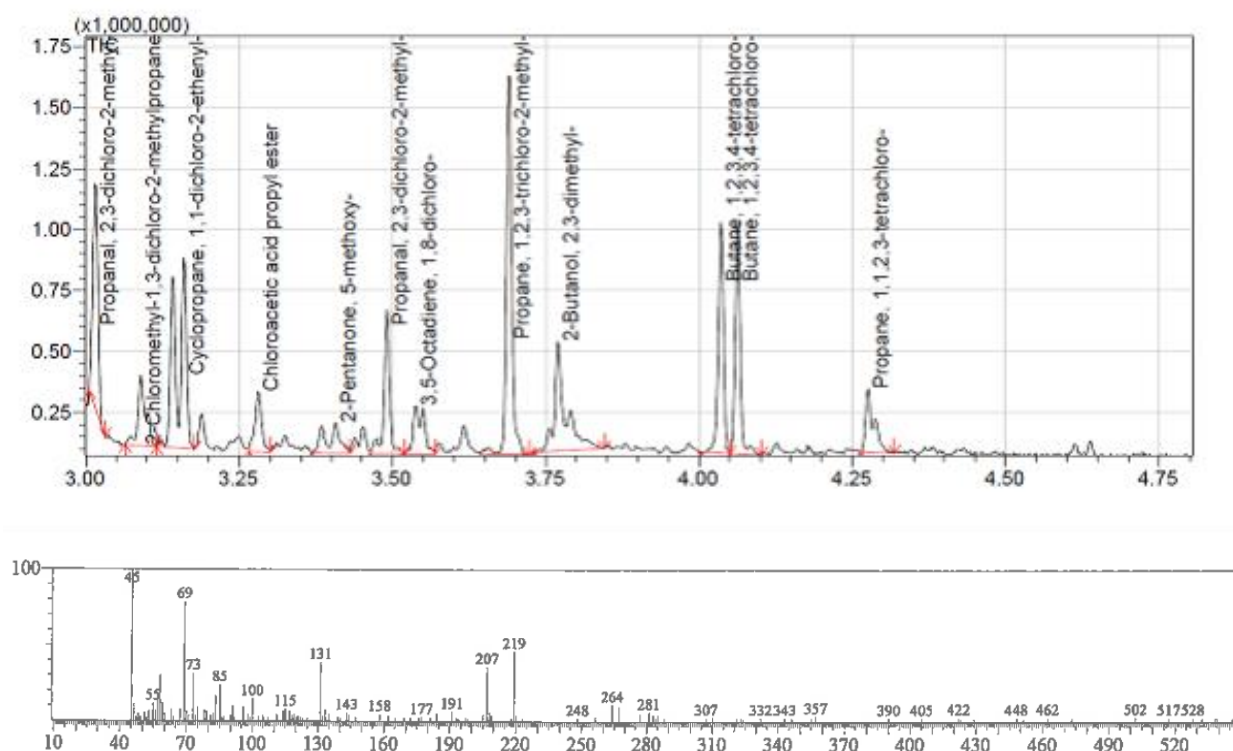


Figure 6. GC-MS chromatograph of NVR from a scavenger plate.

Discussion of Nonvolatile Residue Collection and Analysis Procedure(s)

Nonvolatile residue collected in scavenger plates, Quartz Crystal Microbalance's QCM's, and cold fingers after a bake-out from one of the thermal vacuum chambers located in the SITB at NASA LARC is extracted with 75 ml of isopropyl alcohol according to ASTM E834-81.^{5,6} The extracted samples are delivered to our laboratory located in the Advanced Materials and Processing Branch (AMPB) where NVR measurements are performed to determine the amount (mass) and type of species outgassed from a sample during a bake-out test. The NVR was isolated and analyzed by the following methods and procedures (at AMPB): Mass Determination, FTIR, and GCMS.

Scavenger Plate/Cold Finger Rinse Procedure⁸⁻¹²

Materials:

- Pyrex Bottles
- Isopropyl Alcohol (IPA)
- Gloves
- Safety Glasses
- Anticon Gold® Wipes

- Kapton[®] Tape
- Prepared Sample Labels

General Instructions

1. Obtain one 100 ml Pyrex storage containers (one for each cryogenic surface).
2. Using the predesigned rinse labels found on the shared drive at the following location:
Z:\NVR Analysis\CF_SP Rinse\IPA SAMPLE LABEL.
3. Fill out a label completely and tape to the 100 ml Pyrex container using Kapton[®] tape (they are designed so that 2 inch Kapton[®] tape will cover them completely).
4. Find a clean room approved bottle of Isopropyl alcohol.
5. Follow proper gowning procedure for the cleanroom.
6. Identify the cryogenic surface within the chamber and carefully rinse the full surface of the plate and catch tray with Isopropyl alcohol catching the runoff in the Pyrex container. The runoff will collect in the tray and will drain out the tube located at the bottom. Start rinse at the top of surface and move in horizontal passes. Make sure each pass overlaps the previous and make at least three full rinses of the surface.
7. When there is about 40 ml of rinse in the Pyrex container, the rinse is complete.
8. Place the top on the container and tighten down completely (make sure the cap is on tight so that the IPA does not evaporate).
9. Using an Anticon Gold[®] wipe, clean off the cryogenic surface and catch tray (unless otherwise stated, this can be disposed of in the hazardous waste trash container).
10. Check the travel tote for the following:
 - a. Anticon Gold[®] wipes
 - b. Bottle of IPA
 - c. Gloves (Latex or Nitrile)
 - d. Safety Glasses
 - e. Capillaries
 - f. Sample(s) that are to be tested

11. Bring the travel tote to the Infrared Spectroscopy Laboratory (Building 1293C in room 153; location of FTIR Spectrometer) or the Solution Properties Laboratory (Chemistry laboratory and location of GCMS Spectrometer(s) in building 1293C).

Sample Preparation, Mass Measurement, and Chemical Analysis

The following items are required for NVR analysis:

- Aluminum Weighing Pan(s)
- 100 mL Pyrex Beaker
- Hot Plate
- Bake-out Oven or Furnace (see Figure 7)
- Capillary Tubes
- Scale or Microbalance (see Figure 8)
- Fume Hood
- Desiccator
- Isopropyl Alcohol, spectroscopic grade or better
- Chloroform, spectroscopic-grade or better
- FTIR (Thermoelectron Nexus 670 Spectrophotometer Fourier Transform Infrared Spectrophotometer)
- Gas Chromatograph (Shimadzu GCMS-QP2010 Series Gas Chromatograph and Mass Spectrometer)
- SafeSkin Corp. Hypoclean Powder Free Nitrile Gloves or equivalent brand
- Furnace Gloves
- Laboratory Coat

Safety Precautions and Warnings

- As with any handling of chemicals, do not inhale vapors produced. Chloroform is used in this experiment. These solvents are flammable.
- Wear closed toe shoes in the laboratory.
- Wear long pants in the laboratory.
- Any shoulder length or longer hair should be secured.

General Instructions

Aluminum Weighing Pan Preparation

1. Clean aluminum pans with IPA.
2. Bake-out the aluminum pans at 550°C for at least 1 hour to remove factory coating.

Sample Preparation

1. Rinse the sample from the surface (scavenger plate, cold finger, witness plate, etc.) with about 70 mL IPA.
2. Collect the solvent into a clean glass container. Label and cover tightly to prevent contamination and evaporation during transport.

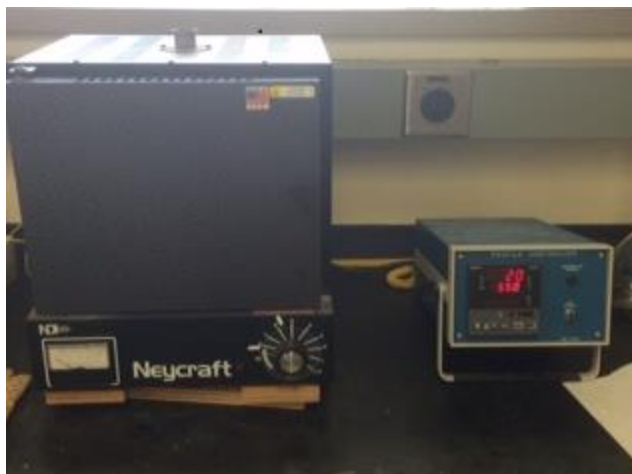


Figure 7. Bake-out Furnace Setup (Building 1293C room 266).

Procedure

1. Take the final mass of aluminum pan to 3 significant figures.*
2. Quantitatively pour collected solvent mixture into aluminum pan while recording the exact volume delivered to the aluminum pan.
3. Heat solution using a hot plate on a low setting to avoid burning off any organics.
4. Evaporate to approximately half of the initial volume, and remove the pan containing the mixture from the hot plate.
5. Cover the mixture in pan with aluminum foil and leave under fume hood overnight.
6. Take a second mass measurement in the morning.*
7. Cover the bottom of the aluminum pan containing mixture with chloroform.
8. Swirl mixture in pan to wet the bottom surfaces.

9. Tilt aluminum pan under fume hood, and let mixture evaporate until about 1 mL remains.
10. Use a capillary tube to place a droplet of solution onto the ZnSe crystal on the FTIR spectrometer (Figure 9).
11. Make solutions with NVR in chloroform in appropriate concentrations in GC ampoules.
*Take a record of the measurement.



Figure 8. Microbalance Setup (Building 1293C room 266).

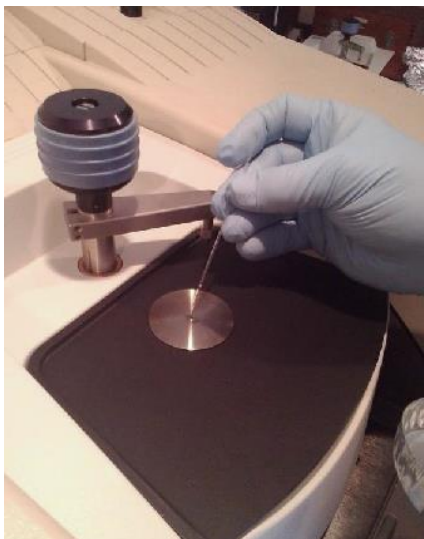


Figure 9. Adding the sample to the ZnSe crystal on the FTIR spectrometer.¹⁷

Flow Diagram (Instructions per day)

Day 1

1. Clean aluminum pans with IPA.
2. Bake-out the aluminum pans at 550°C for at least 1 hour to remove factory coating.

Day 2

1. Rinse the sample from the surface (scavenger plate, cold finger, witness plate, etc.) with about 70 mL of isopropyl alcohol (IPA).
2. Collect the solvent into a clean glass container. Label and cover tightly to prevent contamination and evaporation during transport.

Day 3

1. Take the final mass of aluminum pan to 3 significant figures.
2. Quantitatively pour collected solvent mixture into aluminum pan while recording the volume delivered to the aluminum pan.
3. Heat solution using a hot plate on a low setting to avoid burning off any organics.
4. Evaporate to approximately half of the initial volume, and remove the pan containing the mixture from the hot plate.
5. Cover the mixture in the pan with aluminum foil and leave under fume hood overnight.

Day 4

1. Take a second mass measurement in the morning.
2. Cover the bottom of the aluminum pan containing mixture with chloroform.
3. Swirl mixture in pan to wet the bottom surfaces.
4. Tilt aluminum pan under fume hood, and let mixture evaporate until about 1 mL remains.

Day 4

1. Use a capillary tube to place a droplet of solution onto the ZnSe crystal.
2. Perform FTIR measurement.

Day 5

1. Make solutions with NVR in chloroform in appropriate concentrations in GC ampoules.
2. Perform Gas Chromatography Mass Spectroscopy (GCMS) measurements.
3. Generate report.

Example of Nonvolatile Residue Chemical Analysis Report

**ADVANCED MATERIALS & PROCESSING BRANCH
BRANCH D307
CHEMICAL ANALYSIS REPORT**

TO: Systems Integration and Test Branch/D210

FROM: Advanced Materials & Processing Branch/D307/Dr. Keith Gordon

SUBJECT: 6X6 Characterization Test

DATE: May 9, 2014

ANALYSIS #: 02-6X6

PROJECT: SAGE III-ISS

Samples Submitted:

Sample(s): Facility Scavenger Plate and Cold fingers and Post Test samples
Item: Chamber Background
Facility: 6X6 TVAC

Analysis Performed:

[X] Mass Determination (NASA ECN 1815360)

[X] Fourier Transform Infrared Spectroscopy {FTIR} (NASA ECN 2036845)

[X] Gas Chromatography/Mass Spectrometry {GC/MS} (NASA ECN 1955352)

Results:Facility Scavenger Plate

Total Amount of Residue: 1.65 ± 0.06 mg

Chemical Constituents (*in order of abundance*):

1. Bis(2-ethylhexyl) nonanedioic acid (ester)- plasticizer
2. Various hydrocarbons
3. DC 704 pump fluid
4. Tetratriacontane
5. 1,2,3-trichloro-2-methylpropane
6. 1,2,3,4-tetrachlorobutane
7. DC-705 pump fluid
8. 1,1-dichloro-2-ethynyl cyclopropane
9. 2-ethylhexyl chloroformate

Facility Scavenger Plate Post Test

Total Amount of Residue 0.73 ± 0.06 mg

Chemical Constituents (*in order of abundance*):

1. Various hydrocarbons
2. Bis(2-ethylhexyl) nonanedioic acid (ester) - plasticizer
3. 1,2,3-trichloro-2-methylpropane
4. 1,1-dichloro-2-ethynyl cyclopropane
5. 1,2,3,4-tetrachlorobutane
6. 2,3,-dichloro-2-methyl propanal
7. 2,3-dimethyl-2-methylbutanol
8. 1,3-dichloro-2-2-chloromethyl
9. DC 704 pump fluid
10. 1,1,1-tris(chloromethyl)ethane
11. 1,1,2,3-tetrachloropropane

Facility Cold finger

Total Amount of Residue: 0.03 ± 0.06 mg

Chemical Constituents (*in order of abundance*):

1. 4-chloro-octane
2. 1,2,3,4-tetrachlorobutane
3. 1,2,3-trichloro-2-methylpropane

4. 2,3-dichloro-2-methylpropanal
5. 2,3-dimethyl-2-butanol
6. 1,2,3,4-tetrachloropropane
7. (hexylsulfonyl)-benzene
8. 1,1,3-trichloro-2-propanone

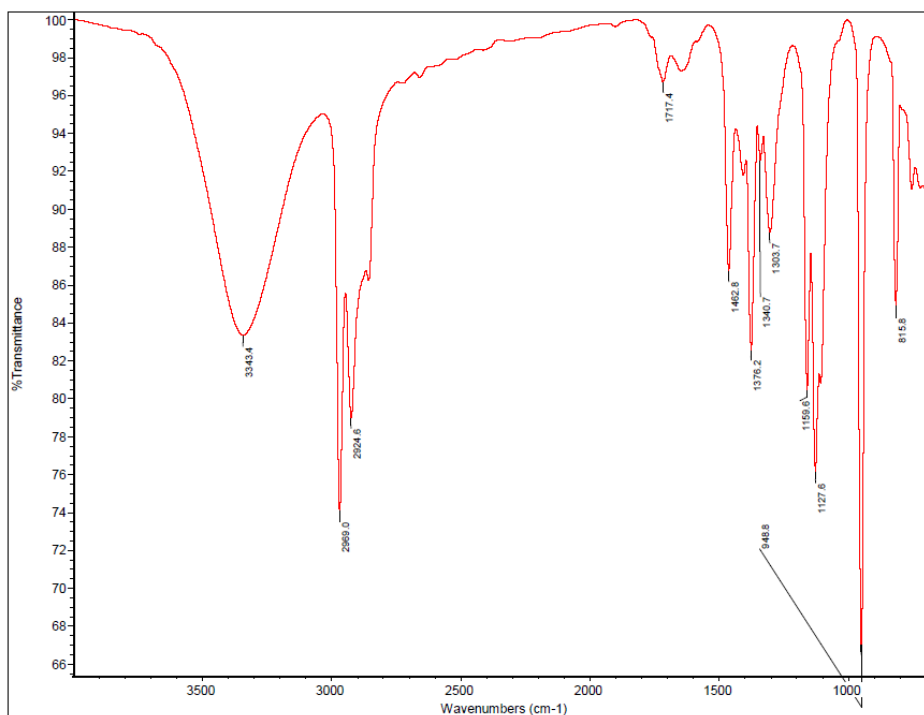
Facility Cold finger Post Test

Total Amount of Residue: 0.32 ± 0.06 mg

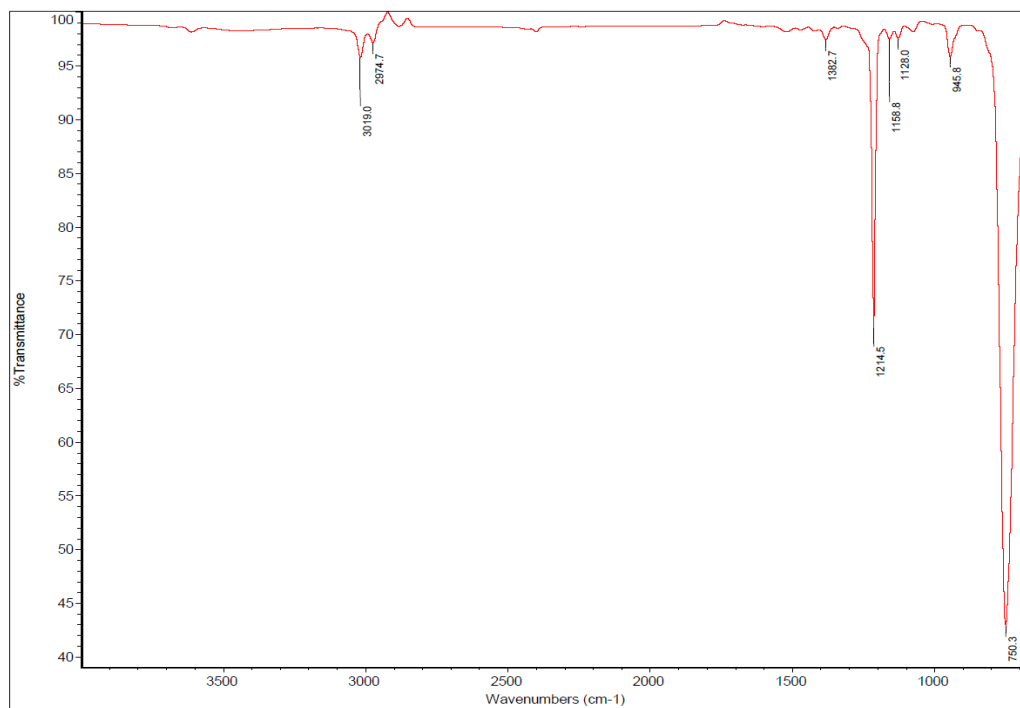
Chemical Constituents (*in order of abundance*):

1. 1,2,3-trichloro-2-methylpropane
2. 1,2,3,4-tetrachloro-butane
3. 1,1-dichloro cyclopropane
4. 2,3-dimethyl-2-butanol
5. 2,3-dichloro-2-methylpropanal
6. 1,3-dichloro 2-chloromethyl
7. 1,8-dichloro 3,5-octadiene
8. Chloroacetic acid propyl ester
9. 5-methoxy-2-pentanone

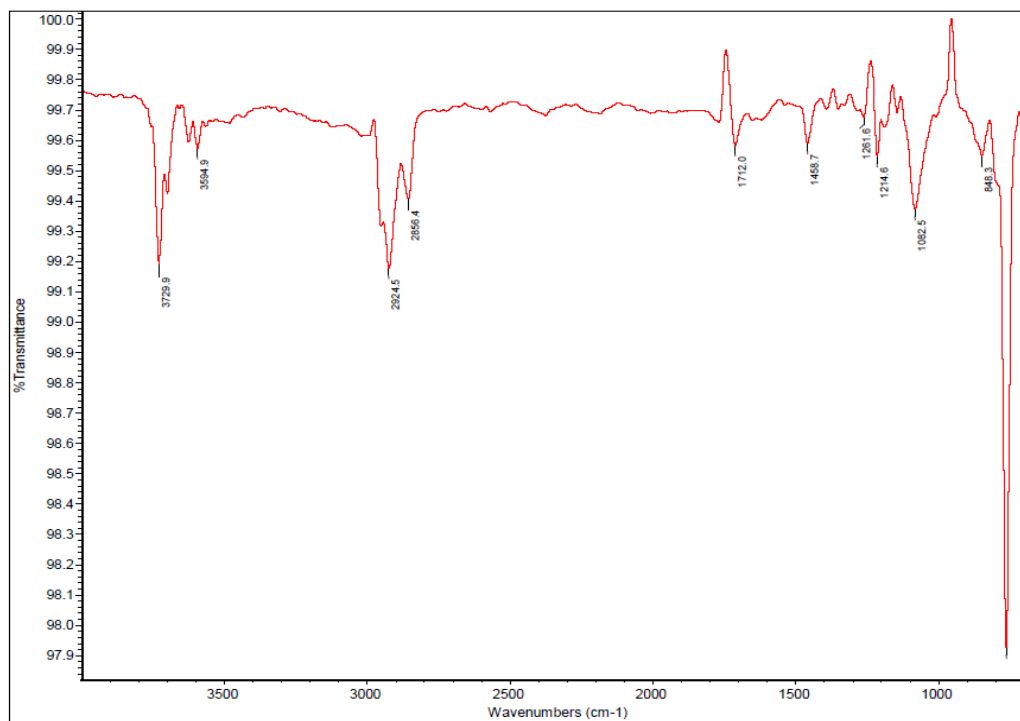
FTIR Results - Scavenger Plate



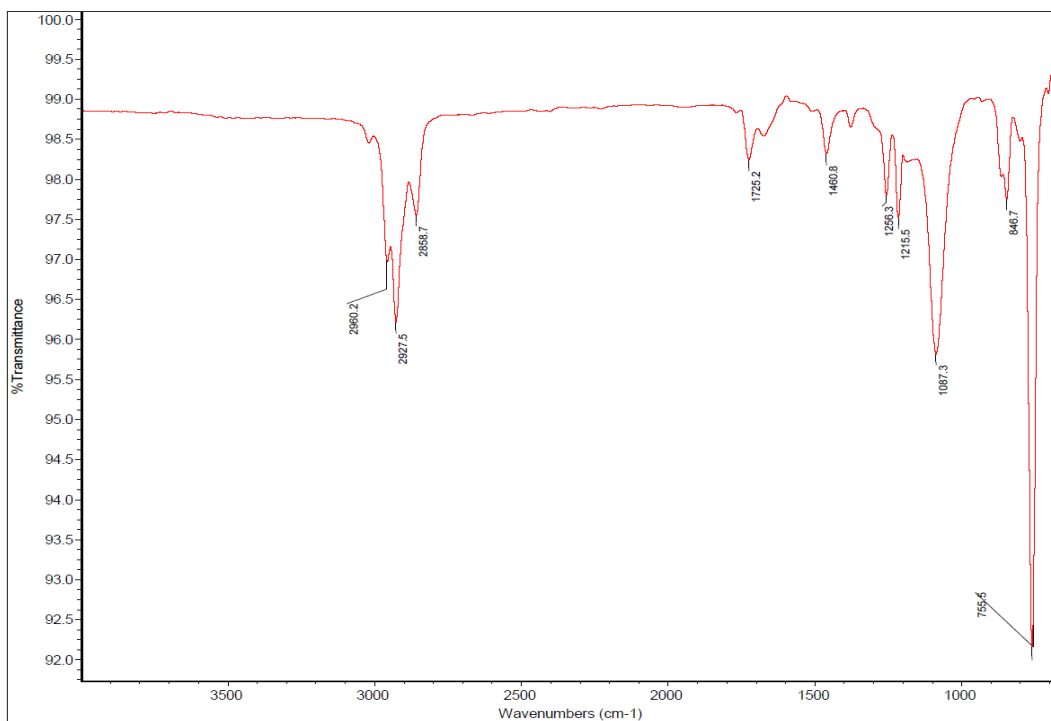
FTIR Results – Scavenger Plate Post Test



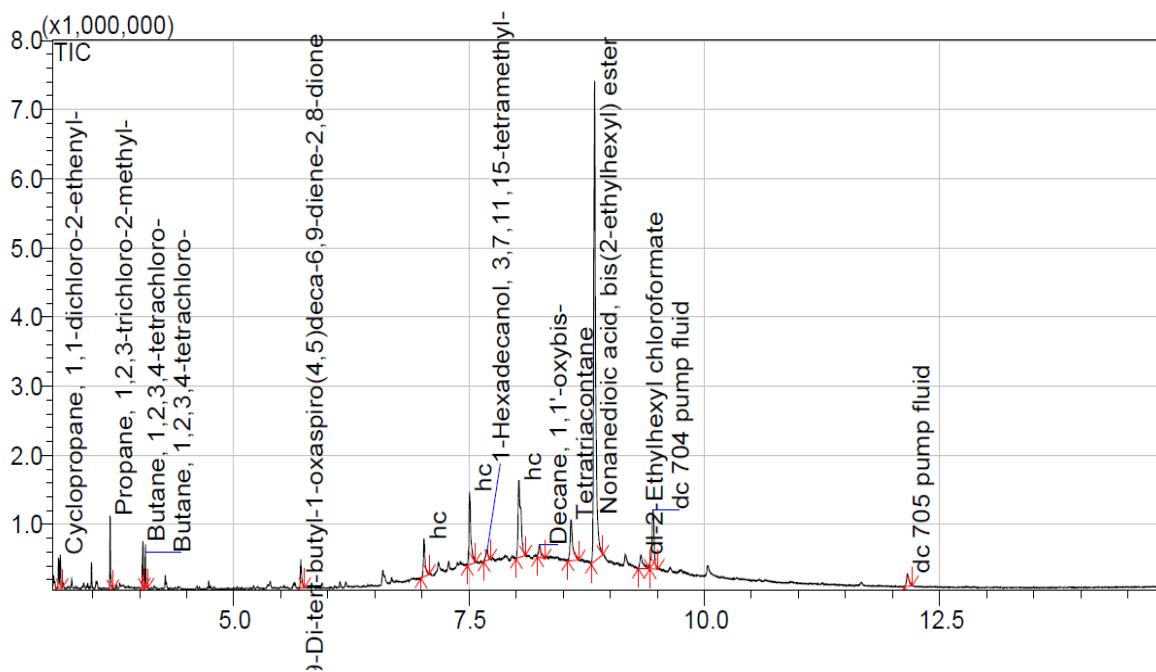
FTIR Spectra –Facility Cold Finger



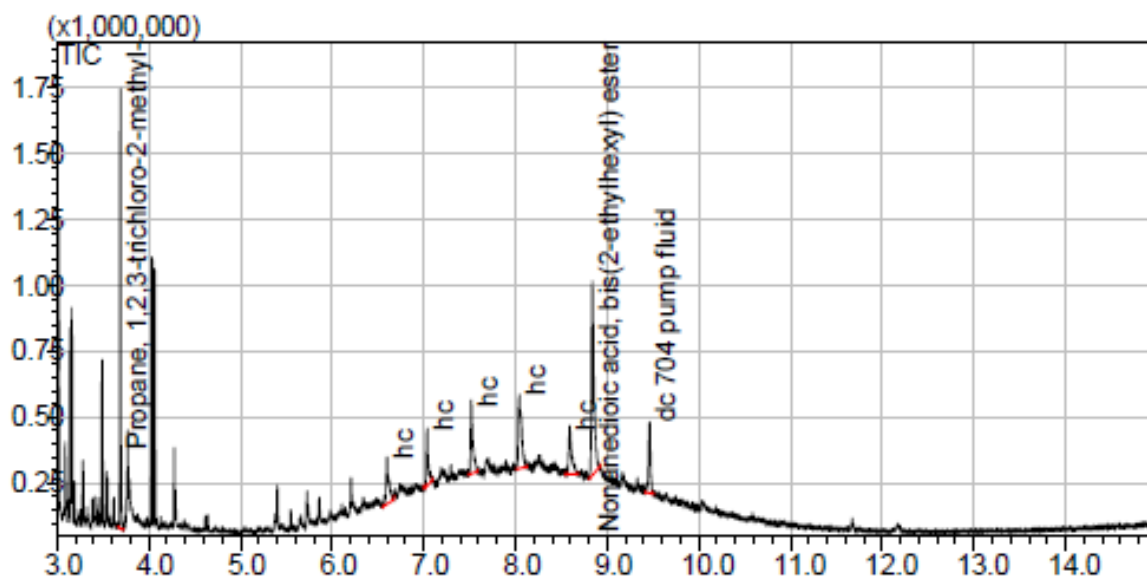
FTIR Spectra –Facility Cold Finger Post Test



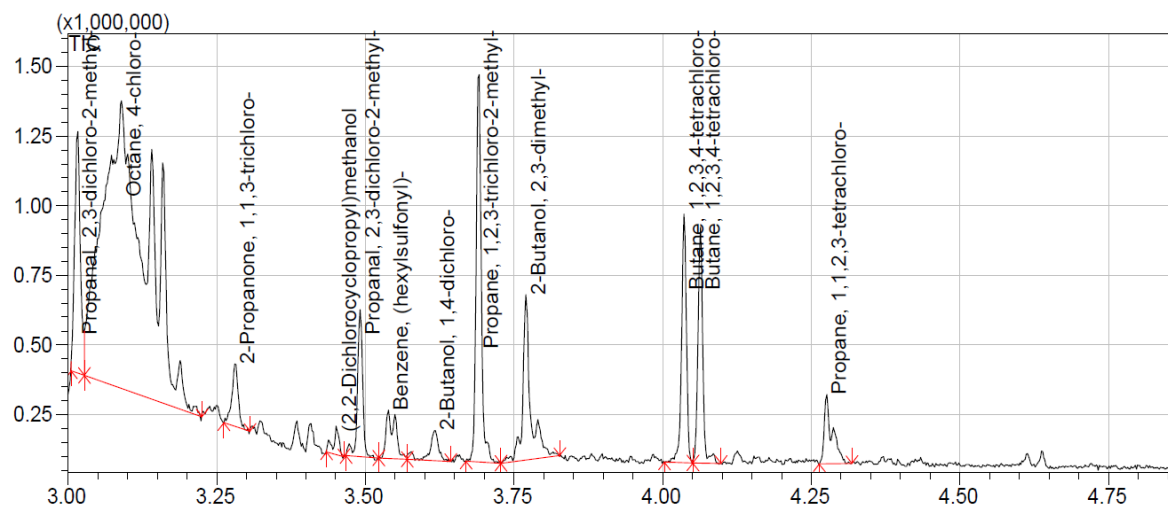
GC-MS Spectra – Scavenger Plate



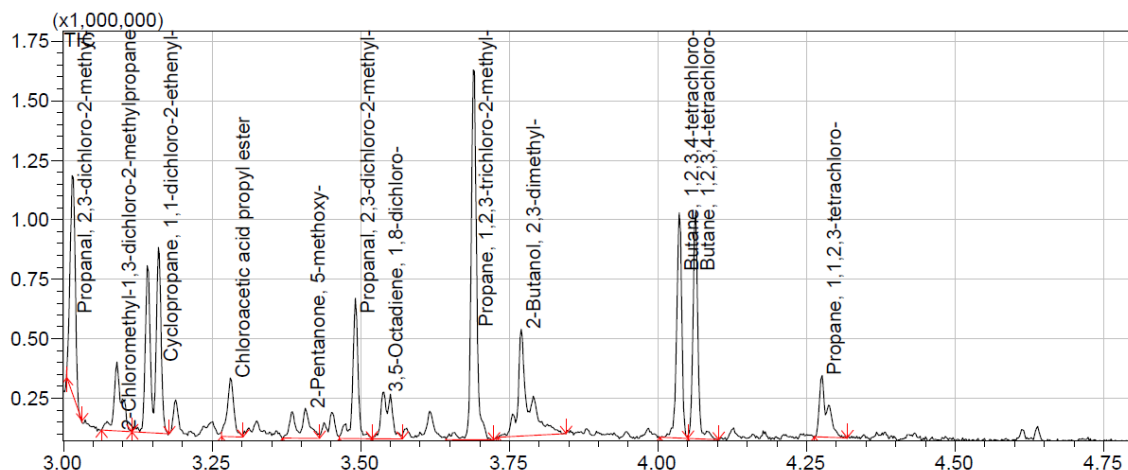
GC-MS Spectra – Scavenger Plate Post Test



GC-MS Spectra – Cold Finger



GC-MS Spectra – Cold Finger Post Test



Comments:

The residue collected by the facility scavenger plate primarily consisted of bis(2-ethylhexyl) nonanedioic acid (ester) and various hydrocarbons. The residue collected by the facility scavenger plate post-test consisted of various hydrocarbons and bis (2-ethylhexyl) nonanedioic acid (ester). The FTIR spectra for the scavenger plate and scavenger post-test are very similar, however an intense OH stretch (3300 cm^{-1}) and $-\text{CH}_2$ stretch (2900 cm^{-1}) are present in the scavenger plate's spectrum and is not present in the post-test's spectrum. The OH stretch is attributed to the structure of the organic molecule, bis(2-ethylhexyl) nonanedioic acid (ester). The difference in spectra is attributed to the ester being present in greater quantity in the scavenger plate than in the scavenger plate post-test sample. Also, please note that GCMS indicated the presence of a small amount of pump fluid in both samples (2-3%).

The residue collected by the facility cold finger primarily consisted of 4-chloro-octane and 1,2,3,4-tetrachlorobutane (both are chlorinated hydrocarbons). The residue collected by the facility cold finger post-test consisted 1,2,3-trichloro-2-methylpropane, 1,2,3,4-tetrachlorobutane, 1,1-dichloro-cyclopropane and 2,3-dimethyl-2-butanol (all, except the last one, are chlorinated hydrocarbons). The FTIR spectra for the cold finger and the cold finger post-test are fairly similar. Two peaks at about 3700 cm^{-1} and 3200 cm^{-1} are present in the cold finger's FTIR spectrum, but are absent in the cold finger post-test's FTIR spectrum. Two peaks are most likely indicative of the presence of aldehyde and/or ketone molecules in the sample(s).

Signed copy available upon request

Keith Gordon

cc:

D210/Aranda,D.

D210/Johnson, T.

D210/Thornblom,M.

D307/ Bryant,R.

D210/O'Connell, J.

D210/Rice, C.

D307/ Edahl, R.

Summary

Nonvolatile residue on spacecraft surfaces can have severe effects on the successful operation of many advanced scientific experiments, therefore the detection and identification of such contaminants are essential.¹ The molecular residue that accumulates on spacecraft components can be isolated via various detection NVR techniques and the total mass can be gravimetrically measured. Chemical analysis of NVR can be accomplished using FTIR, GCMS, or both. Thus, the resulting analysis identifies the components, gives semi-quantitative estimates of contaminant thickness, indicates possible sources of the NVR, and provides guidance for effective cleanup procedures. Additionally, the procedures for collection, determining the mass and chemical make-up of NVR have been outlined and a step-by-step method for the nonvolatile residue analysis process is provided.

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**APPENDICES A and A.1: FOURIER TRANSFORM INFRARED ANALYSIS FOR
CRYOGENIC SURFACE RINSE**

FOURIER TRANSFORM INFRARED ANALYSIS FOR CRYOGENIC SURFACE RINSE

Authored by: Amanda Stark

April 29, 2015

Procedure Overview

PURPOSE:

To provide step-by-step procedures that will allow for a Fourier Transform Infrared (FTIR) Spectroscopy analysis of nonvolatile residue (NVR) contaminants that are present in a cryogenic surface rinse collected after the completion of a thermal vacuum test.

SCOPE:

Fourier Transform Infrared Spectroscopy utilizes a Thermo Nicolet Nexus 670 Spectrometer found in the Advanced Materials & Processing Branch, building 1293C room 153 C. With the use of the Smart iRT accessory, the spectrometer is setup so that a quick analysis can be made of the rinse from a scavenger plate and/or cold finger. The OMNIC software has the capabilities of identifying compounds that have similar spectrum and identify which chemical functional groups are present in the sample based on the absorption peaks in the spectrum.

PERSONNEL:

One (1) qualified FTIR spectrometer operator.

SUPPORT AND SAFETY SERVICES:

Personnel working in the Downflow Cleanroom and in the thermal vacuum chamber will be required to wear approved cleanroom apparel in accordance with test hardware requirements. All personnel using the dedicated chemistry lab must have completed the required safety procedures and all safety standards must be followed. Contact the Facility Safety Head (FSH) for building 1293C for training.

Working with chemicals:

- Chemical Worker DVD
- Hazard Communication
- Annual Waste Management/Spill Response Training
- Personal Protective Equipment PPE Awareness Training
- Document review:
 - Building 1293A/C Chemical Hygiene Plan
 - Lab Specific chemical hygiene plan
- Liquid nitrogen training

Potentially Hazardous Materials PHM permit required:

- Completed form 66 and annual physical

INITIAL CONDITIONS:

A test is completed in one of the vacuum chambers and the cold finger and/or scavenger plate was used.

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List of Acronyms

ATR	Attenuated Total Reflection
FTIR	Fourier Transform Infrared Spectroscopy
FSH	Facility Safety Head
IPA	Isopropyl Alcohol
LN2	Liquid Nitrogen
MCT/A	Mercury Cadmium Telluride/A
NVR	Nonvolatile Residue
PDF	Portable Document Format

SITB	Systems Integration and Test Branch
ZnSe	Zinc Selenide

Useful Images



Figure A1: FTIR equipment setup (Building 1293C room 153C)

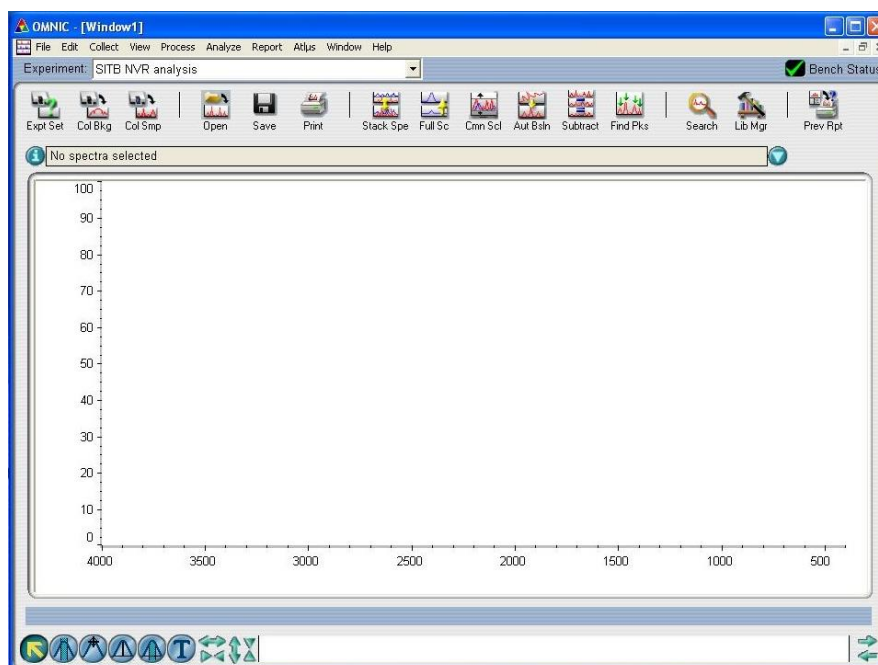


Figure A2: Basic screen for OMNIC software

Scavenger Plate/Cold Finger Rinse Procedure

Materials:

- Pyrex Bottles
- Isopropyl Alcohol (IPA)
- Gloves
- Safety Glasses
- Anticon Gold[®] Wipes
- Kapton[®] Tape
- Prepared Sample Labels

NOTE:

This section of the procedure should be re-evaluated to remove the possibility of contaminating the cryogenic surface rinse!

1. Obtain one 100 mL Pyrex storage containers (one for each cryogenic surface).
2. Using the predesigned rinse labels found on the shared drive at the following location: Z:\NVR Analysis\CF_SP Rinse\IPA SAMPLE LABEL.
3. Fill out a label completely and tape to the 100 mL Pyrex container using Kapton[®] tape (they are designed so that 2 in. Kapton[®] tape will cover them completely).
4. Find a clean room approved bottle of IPA.
5. Follow proper gowning procedure for the cleanroom.
6. Identify the cryogenic surface within the chamber and carefully rinse the full surface of the plate and catch tray with IPA catching the runoff in the Pyrex container. The runoff will collect in the tray and will drain out the tube located at the bottom. Start rinse at the top of surface and move in horizontal passes. Make sure each pass overlaps the previous and make at least three full rinses of the surface.

Note:

The cold finger can be difficult to rinse. Make sure the full surface has been rinsed. It is acceptable to drip a small quantity of IPA within the chamber. Do not miss the top flat surface as well.

7. When there is about 40 ml of rinse in the Pyrex container, the rinse is complete.
8. Place the top on the container and tighten down completely (make sure the cap is on tight so that the IPA does not evaporate).
9. Using an Anticon Gold[®] wipe, clean off the cryogenic surface and catch tray (unless otherwise stated, this can be disposed of in the hazardous waste trash container).
10. Check the FTIR travel tote for the following:
 - a. Anticon Gold[®] wipes
 - b. Bottle of IPA
 - c. Gloves (Latex or Nitrile)
 - d. Safety Glasses
 - e. Capillaries
 - f. Sample(s) that are to be tested
11. Bring the travel tote to Infrared Spectroscopy Laboratory (Building 1293C room 153, location of FTIR Spectrometer) or chemistry lab (room 266 in building 1293C).

Sample Preparation and Mass Measurement

NOTE:

This section of the procedure should be re-evaluated so that the full mass of the NVR is found during the Mass Measurement!

1. Clean Aluminum pans with IPA.
2. Bake-out Aluminum pans at 550° C for at least 1 hour to remove coating.
3. Take final Aluminum pan weight.
4. Pour the whole rinse sample into aluminum pan and rinse the bottle with IPA and pour into aluminum pan.
5. Heat solution using a hot plate (low setting to avoid burning off organics).

NOTE:

Only use pre-baked metal dish held in the controlled cabinet. Before adding chloroform to the NVR sample move on to the “Startup Procedure”.

6. Evaporate to approximately ½ volume, remove solution from hot plate.
7. Tent with aluminum foil and leave solution under fume hood overnight.
8. Weight the pan a second time once the IPA is evaporated.
9. Re-dissolve solution with chloroform (enough to cover bottom of Al Pan).
10. Swirl around and wet the bottom surfaces.
11. Tilt aluminum pan and carefully carry it to the FTIR spectrometer.
12. Wait until about 1 ml remains when the pan is tilted.

Startup Procedure

Materials:

- Liquid Nitrogen (LN2)
- Large dewar filled with LN2
- Small dewar
- Thermal gloves
- Safety glasses

NOTE:

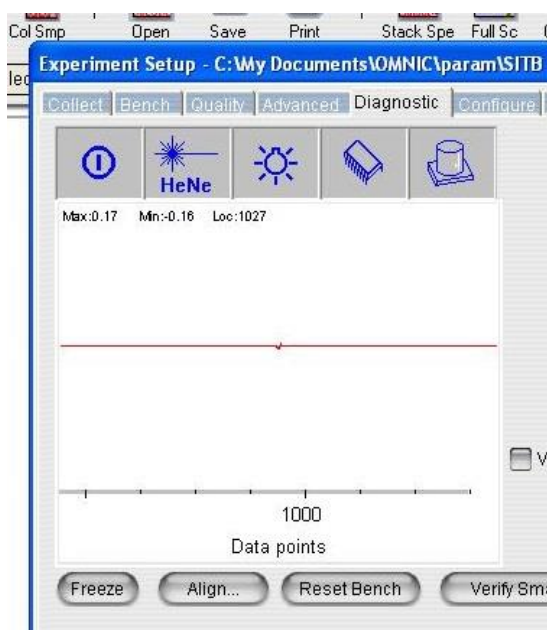
Contact Keith Gordon (phone: 757-864-2201) in the Advanced Materials & Processing Branch so that LN2 is made available. Begin this process prior to adding chloroform to the NVR sample!

1. Log into the FTIR computer using the SITB user name and password:

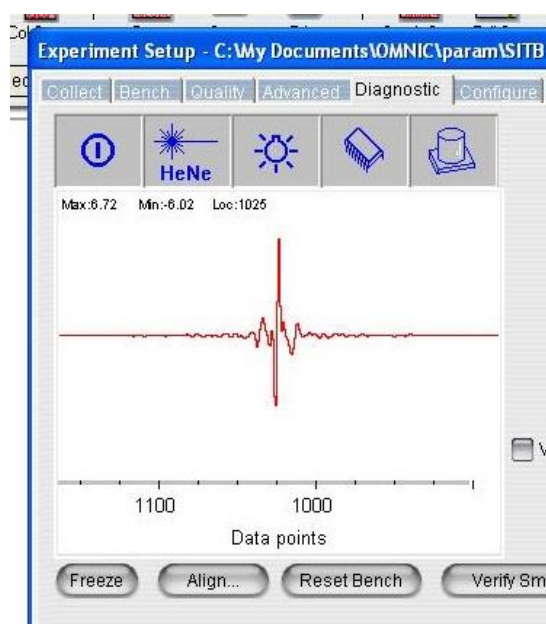
Username: Systems I&T Branch

Password: XXXXX

2. Take the large LN2 dewar and pour LN2 into the small dewar until full. If the Experiment Setup window is open and set to the “Diagnostic” tab, the reaction of the detector can be seen by the increased intensity of the interferogram, (Figure A3):
 - a. To open this window go to “Setting Up a Method” (page 63) in Appendix A.1 and follow steps 1 and 5.



(a)



(b)

Figure A3. (a) Interferogram in “Diagnostic” window before LN2 is added, (b) Interferogram in “Diagnostic” window during LN2 addition.

3. Open the cap located on the top left side and remove the stopper. This is the cap that is in the back, not the front cap that is labeled “DO NOT OPEN!” (Figure A4).



Figure A4. LN2 fill cap.

4. Use the shorter of the two funnels that are located on top of the microscope (Figure A5). Chill the metal tube in the LN2 that is in the smaller dewar and insert it into the fill hole.

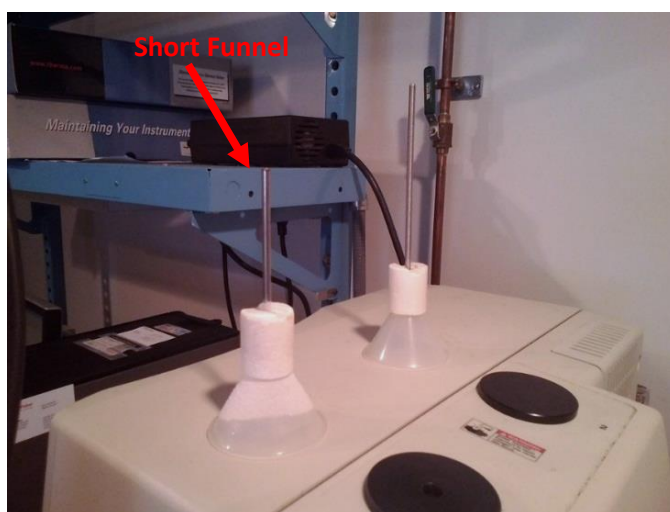


Figure A5. Two funnels on the microscope.

5. Carefully pour the LN2 into the funnel until full. Allow the LN2 to run into the spectrometer. Repeat until the LN2 has overflowed slightly into the well around the funnel.
6. The spectrometer will be ready to be used in 30 minutes and can be used for 19 hours:
 - a. If waiting longer than 30 minutes, more LN2 may need to be added.
7. Remove funnel, replace stopper, and close cap down as much as possible (do not force closed. Depending on the amount of LN2 overflow it may not close at all).

Preparing the Smart iTR

Materials:

- Capillaries
- Latex or Nitrile gloves
- IPA
- Anticon Gold Wipes

NOTE:

This Procedure is primarily written as if the Zinc Selenide (ZnSe) crystal is being used, it can also be used with the Diamond crystal assembly if the Experiment Method is changed to accommodate it.

1. ZnSe crystal is stored in Solutions Properties Laboratory (room 266) in the “NVR FTIR” drawer (Figure A6). You must also use the Smart iTR platform that is in the same drawer.



Figure A6: ZnSe Crystal (Solutions Properties Laboratory)

2. With gloves on, remove the current Smart iTR from the spectrometer by lifting from the back and front (Figure A7). Carefully place it on the counter since it can be damaged easily.

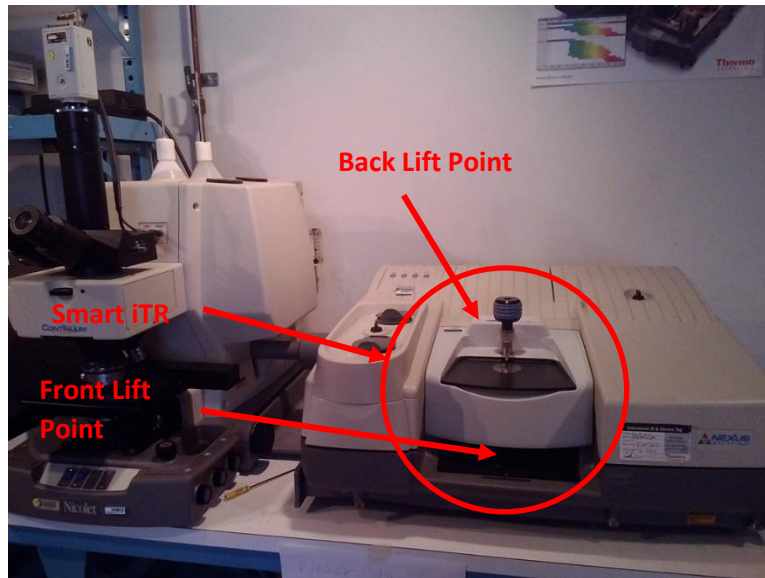


Figure A7. Nexus 670 and continuum microscope

3. Rotate the tip clear of the crystal and dark gray plate (Figure A8).



Figure A8. Tip arm partially rotated out of the way.

4. Remove the dark gray plate and press the crystal assembly clear of its opening (Figure A9). The crystal needs to be pushed from the front to the back. Avoid touching the crystal.
5. Remove the tip by unscrewing from arm:
 - a. There are three different tips, check to see which is currently on the Smart iTR.
 - b. Use the flat tip, it might be in the tip storage found under the gray plate (Figure A10).
6. Clean the dark gray plate, tan surface where the dark gray plate sits and the ledge behind. Also, clean the tip with IPA and Anticon Gold[®] wipes. Be careful to not get any of the IPA on the mirrors located under the crystal.

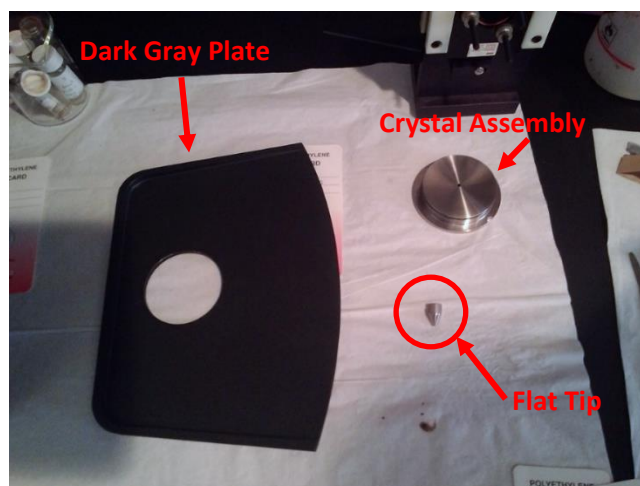


Figure A9. Dark gray plate, flat tip, and crystal assembly.

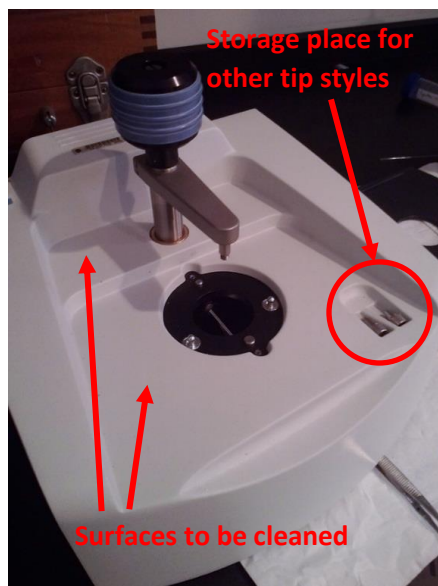


Figure A10. Tan body of the Smart iTR with the dark gray plate removed.

CAUTION!

Do not apply IPA directly on the crystal. Get the Anticon Gold[®] wipes saturated with IPA and then wipe clean.

7. Carefully clean the metal surfaces of the crystal and then the crystal itself.
8. Replace the crystal assembly by fitting it into the plate going from the back to the front. It needs to sit parallel with the top of the surface. The rubber gasket should be seen completely above the surface (Figure A9). Take note about the location of the notches (Figures A12 and A13). They need to fit to the corresponding size peg on the body of the Smart iTR.

NOTE:

When pushing the crystal into the plate only touch the outer ring, see Figure A12.

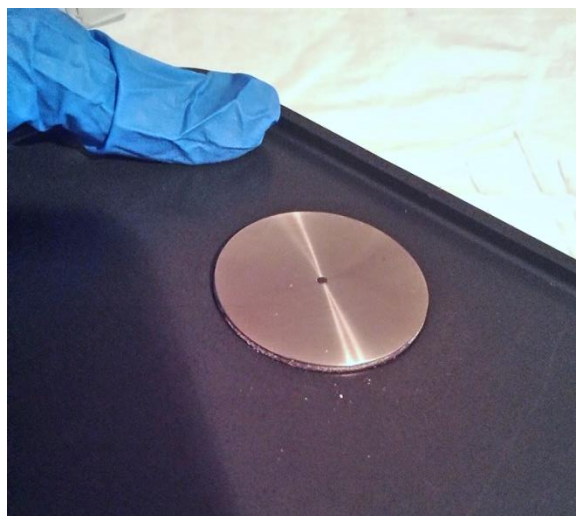


Figure A11. Crystal assembly fitted into dark gray plate.

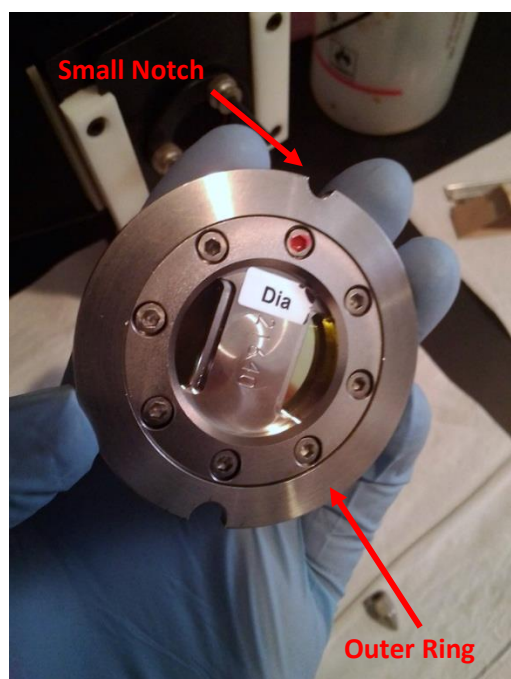


Figure A12. The bottom side of the crystal assembly.

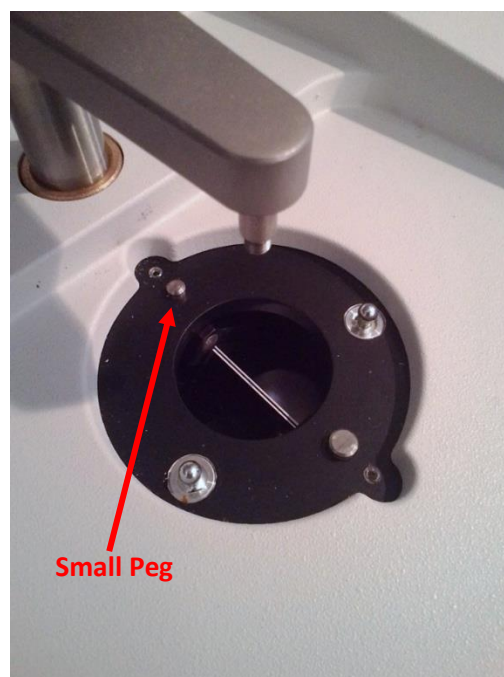


Figure A13. Recessed area for the diamond crystal assembly.

9. While supporting the crystal from the back, carefully place the dark gray plate back on the Smart iTR body while aligning the notches and pegs on the crystal.
10. Replace the tip (the arm can either be left off to the side or moved back over the crystal).
11. Place the Smart iTR into the spectrometer.

Obtaining FTIR Analysis

Materials:

- Thumb Drive

NOTE:

When completed with analysis of all samples, log usage into log book at the computer. This requires date, your name, number of samples, and “Main” or “Microscope”.

1. With the Smart iTR in place, the crystal cleaned, and having filled with LN2 30 minutes prior, open the software.
2. Open the OMNIC software. When the program starts there will be a window indicating which accessory is currently installed. Verify that it is identifying the Smart iTR Accessory and hit “OK” (Figure A14).

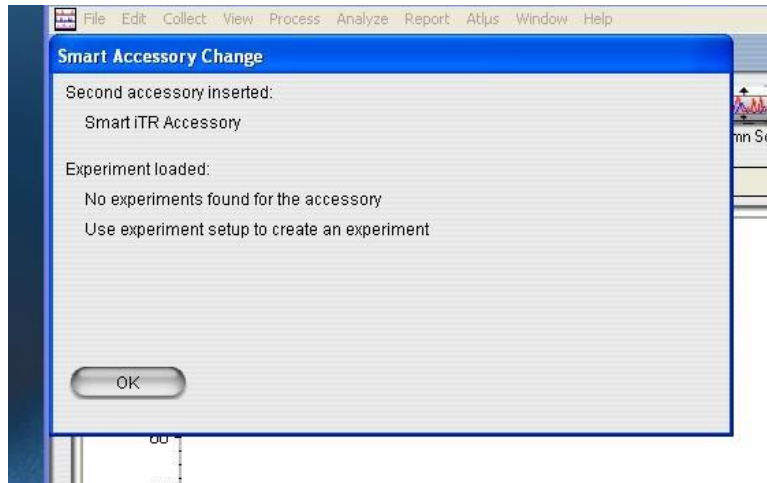


Figure A14. Start-up screen with accessory verification.

3. Check to see if the correct sample method is selected across the top of the screen (Figure A15, to setup a new method see Appendix A.1).

NOTE:

If the accessory does not match the Experiment Method a warning will appear. This seems to be a normal occurrence.

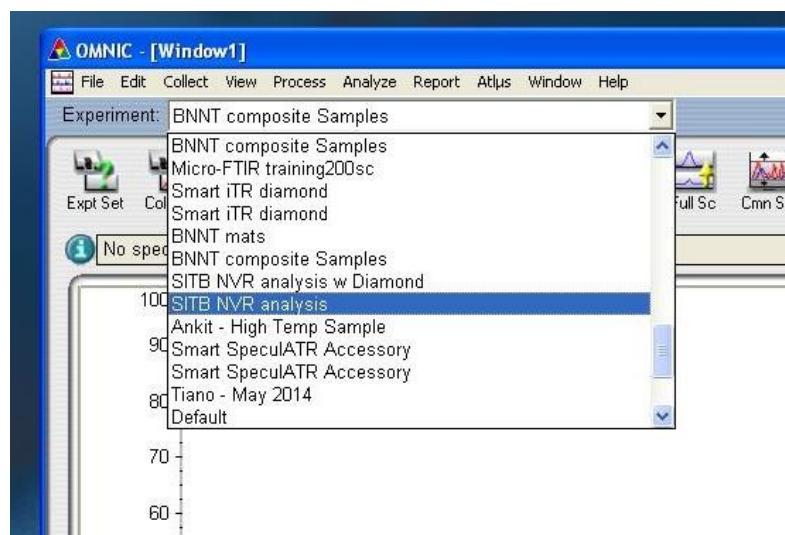


Figure A15. The environment (method) selection bar.

4. Swing the arm with the flat tip away from the crystal (Figure A8).
5. Click on the “Col Smp” and take a background reading (Figure A16).

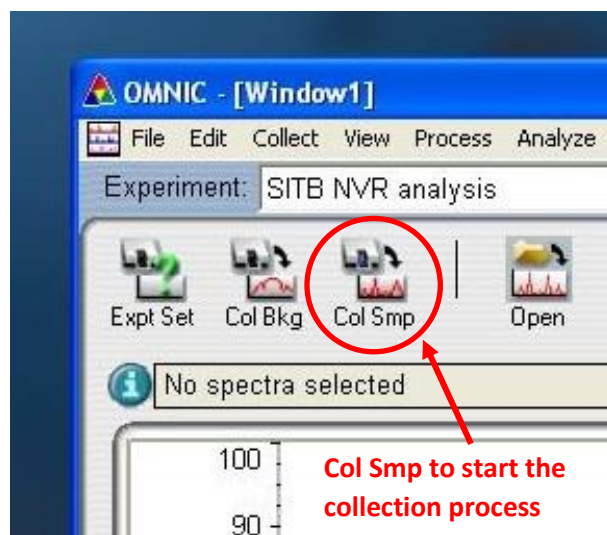


Figure A16: Bar across OMNIC main screen.



Figure A17. Typical background for the ZnSe crystal.

6. A confirmation window will appear, hit "OK" (Figure A18).

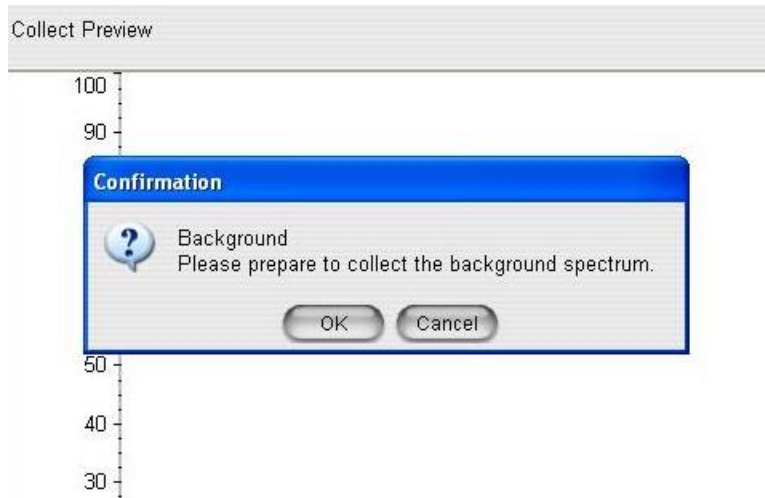


Figure A18: Confirmation window for Background collection.

7. Wait until the initial spectrum is displayed and then click on "Start Collection" in the top right corner (Figure A19). The system will adjust the spectrum based on the auto gain. This will happen until the sample is collected.

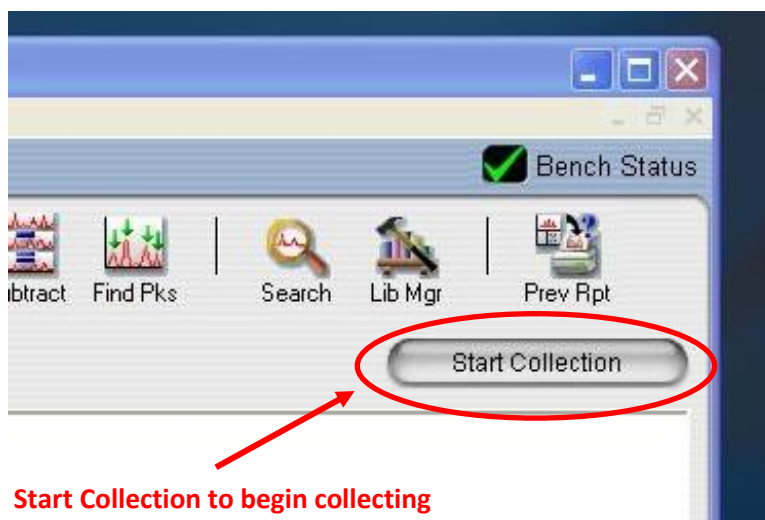


Figure A19. Top right corner of OMNIC main window.

8. A confirmation window will pop up asking if the user wants to take the sample reading, do not hit “OK” until the sample is in place (Figure A20).

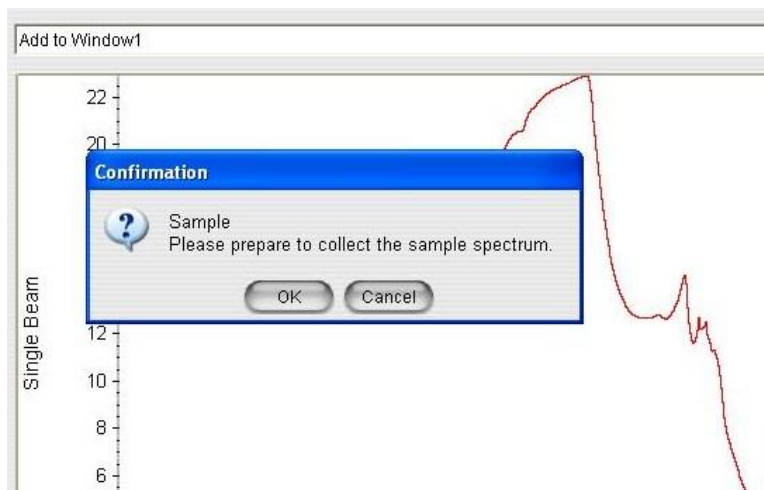


Figure A20: Confirmation window for reading sample.

9. Take a capillary and collect a small amount of test solution, stop off the open end with a finger.
10. Hold the capillary at an angle perpendicular to the crystal surface and allow one drop to settle on the crystal (Figure A21).
11. Wait for the solvent to evaporate.

For Evaluation of Solids Only!!

Swing the arm back over the crystal and turn the blue knob until the flat tip is resting on the crystal (Figure A22).

CAUTION!

Do not over tighten! This will break the crystal. The tip just needs to come in contact with the crystal surface to remove any air pockets between the sample and the crystal.

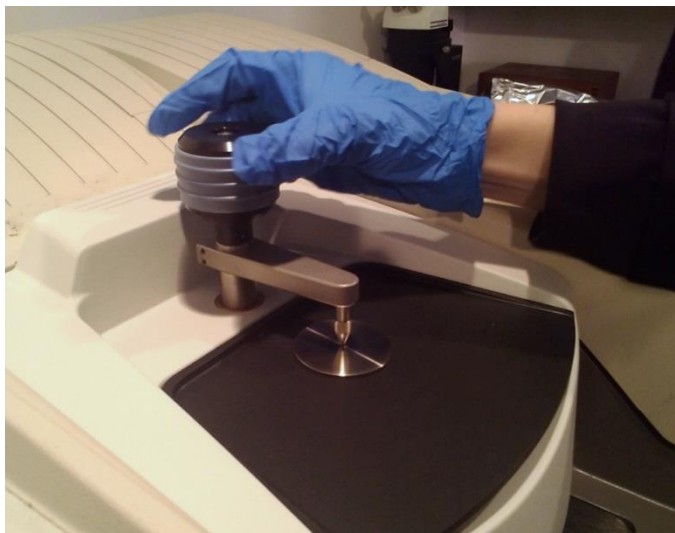


Figure A21. Rotate the blue knob until the tip is touching the surface of the crystal.

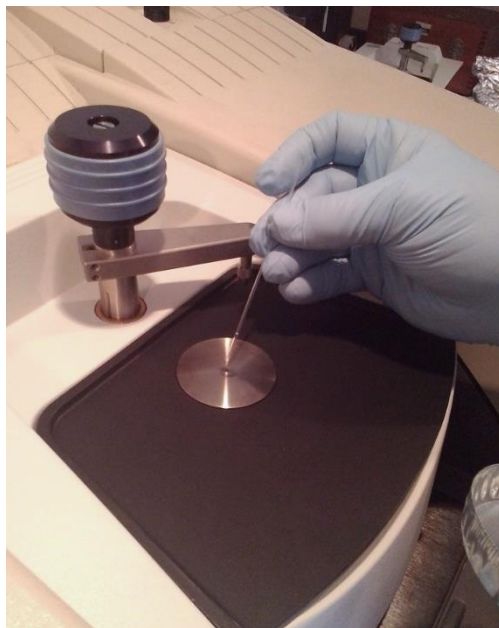


Figure A22. Adding the sample to the crystal surface.

12. Click the “OK” button on the message screen on the computer and wait until the initial analysis appears.
13. Click “Start Collection” on the upper right hand side of the screen and wait for all the samples to be taken (Figure A19).
14. A “Collect Sample” screen will appear and a time stamp will be filled in. Remove the “(GMT-##:##)” from the end and add the name of the spectrum, click “OK” (Figure A23).



Figure A23. Collect Sample Window.

15. Once all the samples have been taken, a confirmation window will appear. Answer “Yes” to the “Add to Window 1” question. (Figure A24).The unprocessed spectrum will appear in the main window (Figure A25).

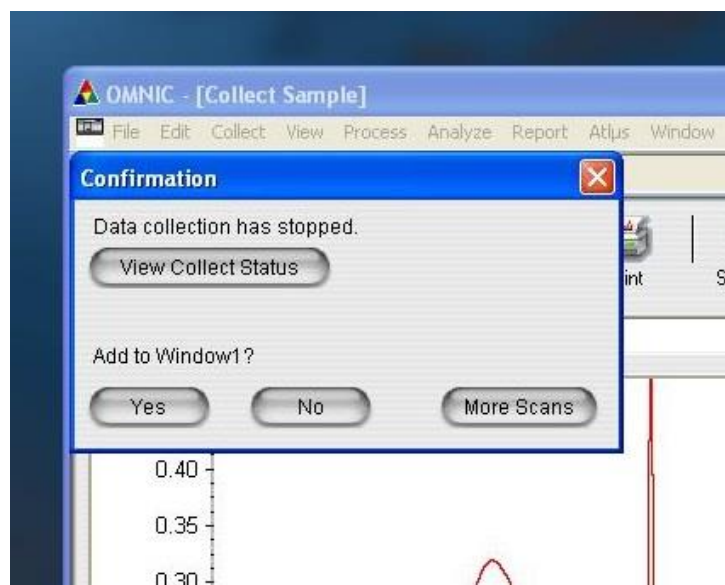


Figure A24: Confirmation window to add the spectrum to the main window.

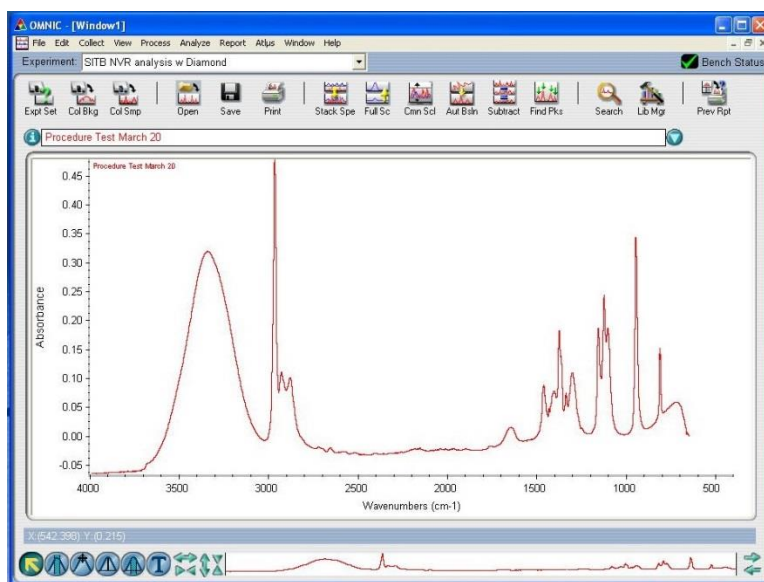


Figure A25. The unprocessed spectrum for IPA using the Diamond Crystal.

Processing and Analyzing the Collected Sample

1. If the grid is not being displayed it, can be found under View → Display Grid (Figure A26).

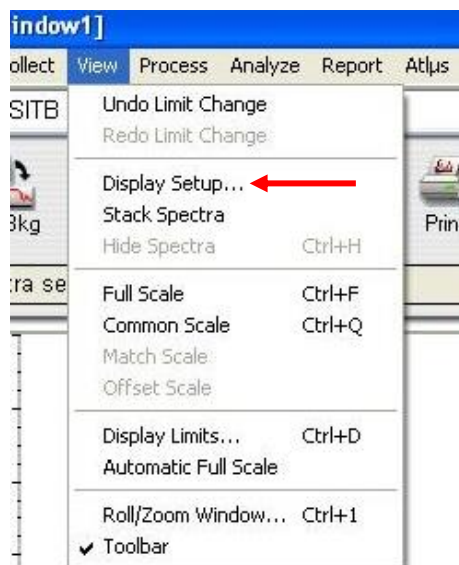


Figure A26. “View” tab options.

2. Check the box for “Show Grid” when the window opens (Figure A27).

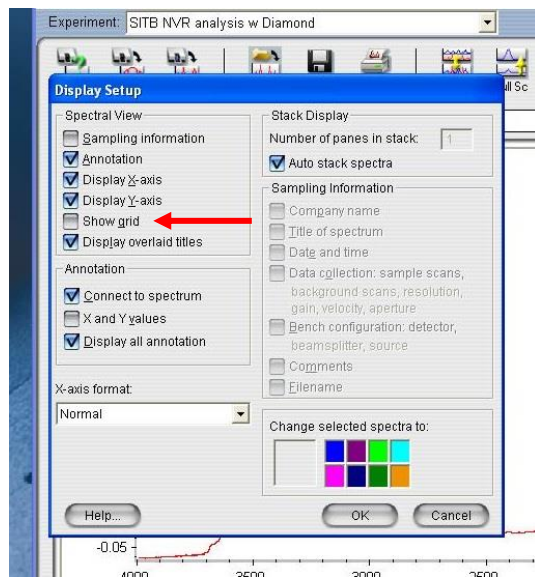


Figure A27. “Display Setup” Window.

3. From the top of the screen, select the “Process” tab (Figure A28):

NOTE:

The next two steps (3A and 3B) require the spectrum to be in Absorbance mode.

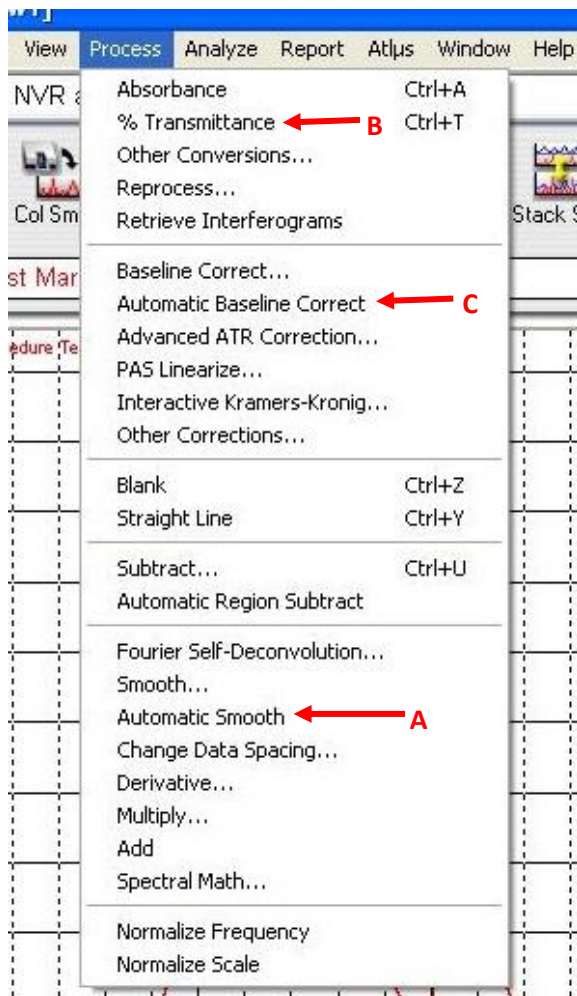


Figure 28. “Process” tab options.

A. The choose “Automatic Smooth”, click on the original spectrum (should turn red) and hide it, View → Hide Spectra.

NOTE:

The original spectrum will appear as a different color while the new spectrum will be red. In the upper right hand corner, there will be two names, one will have a different number of “*” to indicate how far off of the initial spectrum it is (Figure A28). This will happen for both the Automatic Smooth and Baseline Correct Processing steps (Figure A29).

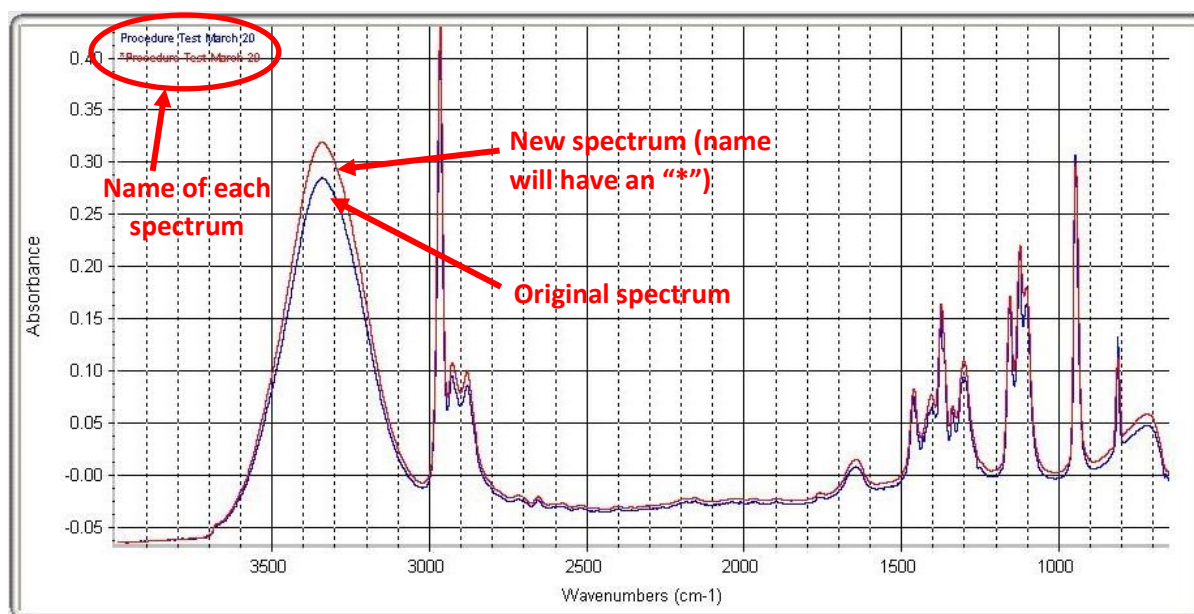


Figure A29. Original and Smoothed Spectra.

- B. Choose “Automatic Baseline Correct”, hide the spectrum with only one “*” (see note under point ‘A’ in this section).
- C. In the “Process” tab, select “% Transmittance” (The “Absorption” option just above this will switch it back if needed).
4. Under the “Analyze” tab select “Find Peaks” (Figure A30).

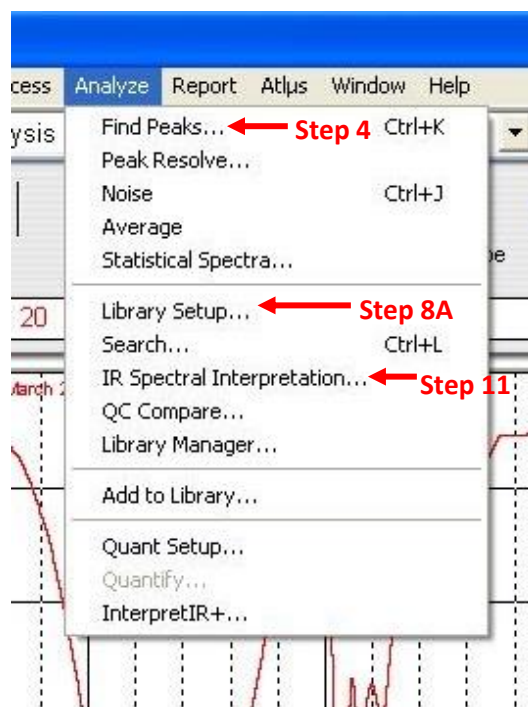


Figure A30. “Analyze” Tab Options.

5. Selecting along the baseline for the spectrum will move the horizontal line to the top of the graph. This will include the most amount of peaks that will be used for analysis. (Figure A31, Figure A32).

NOTE:

If the spectrum has a lot of “noise” the top of the graph may include too many peaks. For a noisy spectrum, a middle area may be a better choice to place the peak line.

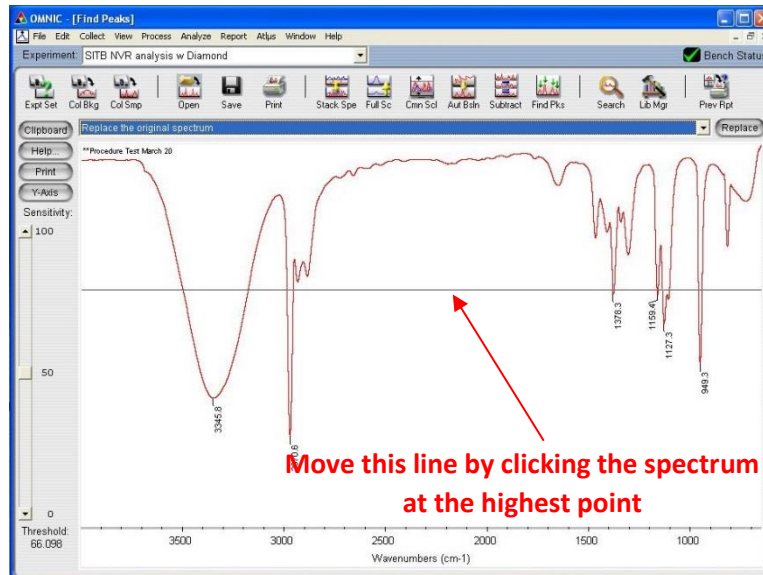


Figure A31. Initial horizontal line position when Automatic peak finding is opened.

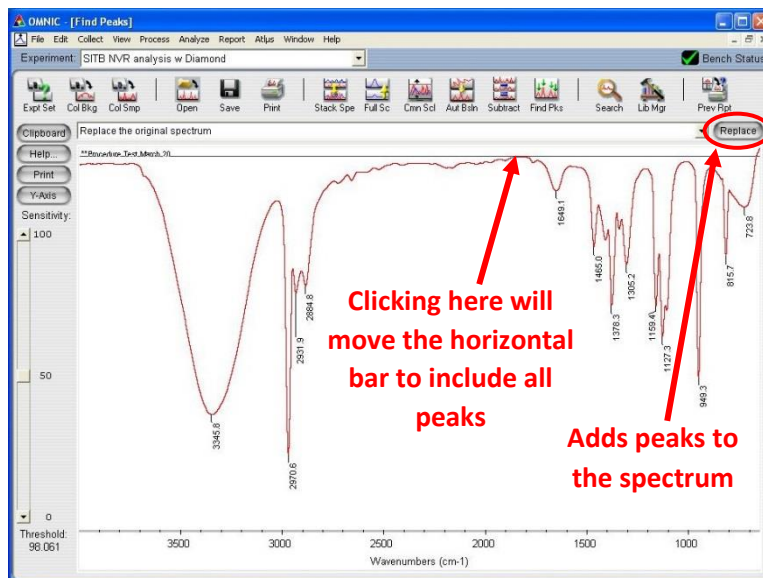



Figure A32. Find Peak line moved to include all peaks.

6. Return to the main screen with the peaks identified by selecting the “Replace” button.
7. The second option for identifying peaks is to do it manually.

Manual Peak Identification

1. Find the  at the bottom left of the screen and select it.
2. Go to any peak to be identified.
3. Click, hold, and drag down on the peak. A box will appear with the wavenumber (Figure A33).

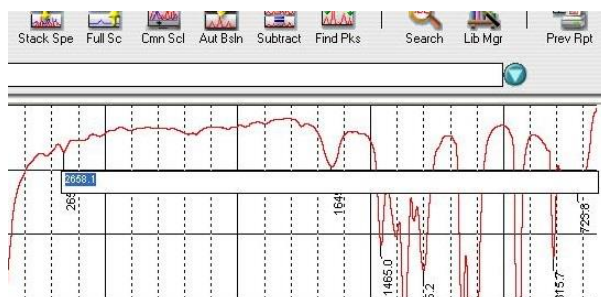


Figure A33. Adding a peak manually.

4. Hit enter.
5. Switch back to the standard cursor, , in the bottom left of the screen.

8. After reviewing the results, the search result window can be closed by selecting the “Close” button in the bottom right corner (Figure A34).



Figure A34. Library search result page.

9. Choose “IR Spectrum Interpretation” to display the groups that are present (Figure A35):
 1. Analyze → IR Spectrum Interpretation (Figure A30).

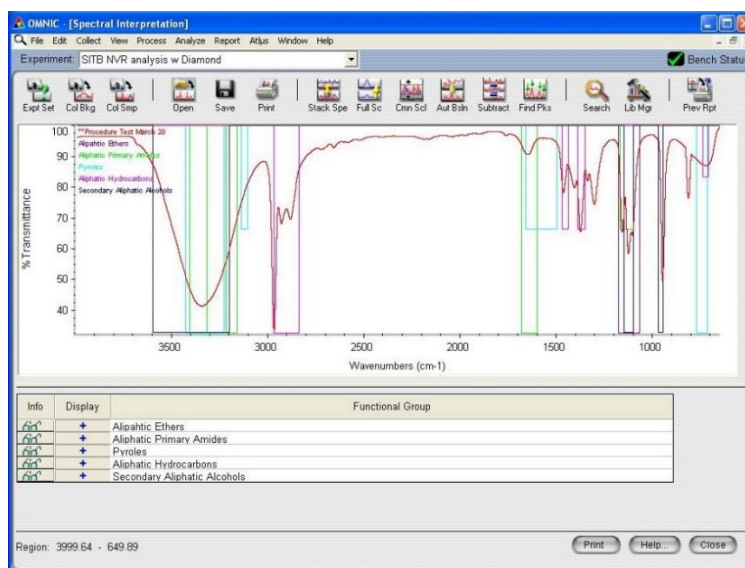


Figure A35. Results of present groups.

10. Print as a PDF by clicking on the print icon in the top horizontal bar.

Saving a PDF Document

1. Print as a PDF by clicking on the print icon in the top horizontal bar.
2. Choose “Printer Setup...” and then the PDF option from the printer drop down menu (Figure A36).

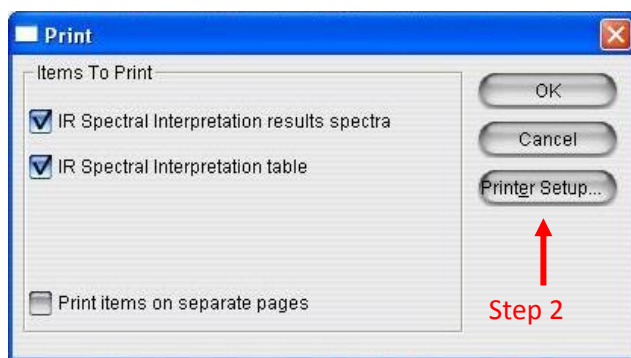


Figure A36. Print screen.

3. Hit “OK” and browse for the SITB file (Figure A37).
 - i. Follow: Local Disk(C:) → My Documents → OMNIC → Spectra → SITB.



Figure A37. Save PDF file window.

4. Change the name so that it includes an indicator that describes the purpose of the file.

5. Hit “OK” to return to the “Save PDF file” window, and then hit “OK” again:
 - i. If the “Open PDF in reader” box is selected, the PDF will appear in Acrobat.

11. Rotate the blue knob until the tip is no longer on the crystal.
12. Clean the crystal and the metal surface using Anticon wipes and IPA. This may have to be done more than once depending on the contamination present in the sample.
13. When completely finished, return the Smart iTR and ZnSe crystal to Solutions Properties Laboratory (room 266). Place the second Smart iTR with the diamond crystal in the spectrometer and log out of the computer.

NOTE:

When completed with all samples, log your usage in the log book at the computer. This requires date, name, number of samples, and “Main”.

14. Close the IR Spectrum Interpretation screen by clicking on the “Close” button in the lower right corner.
15. Save the spectrum as a *.SPA file so that it could be analyzed later.
 - A. Go to: File → Save As.
 - i. The Save As screen will appear, check to make sure the “SITB” file is selected (“**Error! Reference source not found.**” will appear if incorrect file is selected).
 - B. Select “Set File Name to Title” (Labeled as “B” in Figure A38).

16. Remove the “@@” from the title, and save.

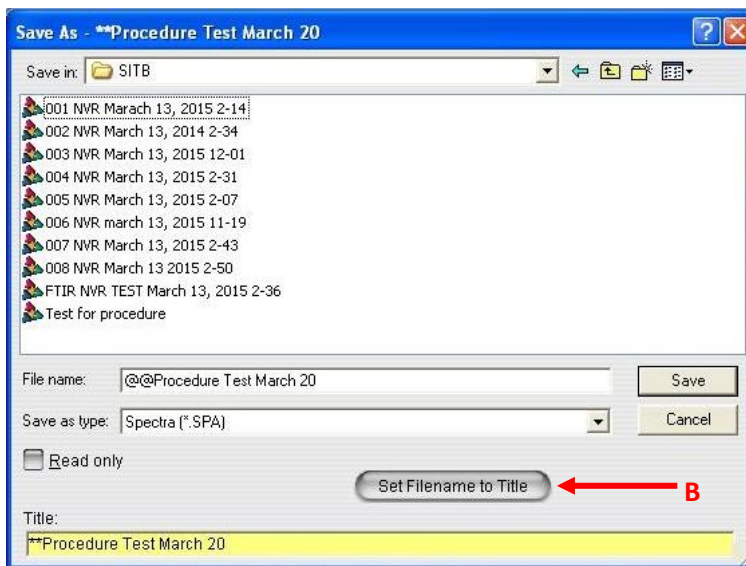


Figure A38. “Save As” Screen for *.SPA files.

Appendix A.1

NOTE:

This section (Appendix A.1) is only needed if another method of collection is required. The “SITB NVR Analysis” has been setup for use for scavenger plate and cold finger analysis. Any of the settings can be changed. The following are the settings chosen for the SITB contamination evaluations.

Setting Up a Method:

1. Select “Expt Set” from the bar across the top of the screen (Figure A39).

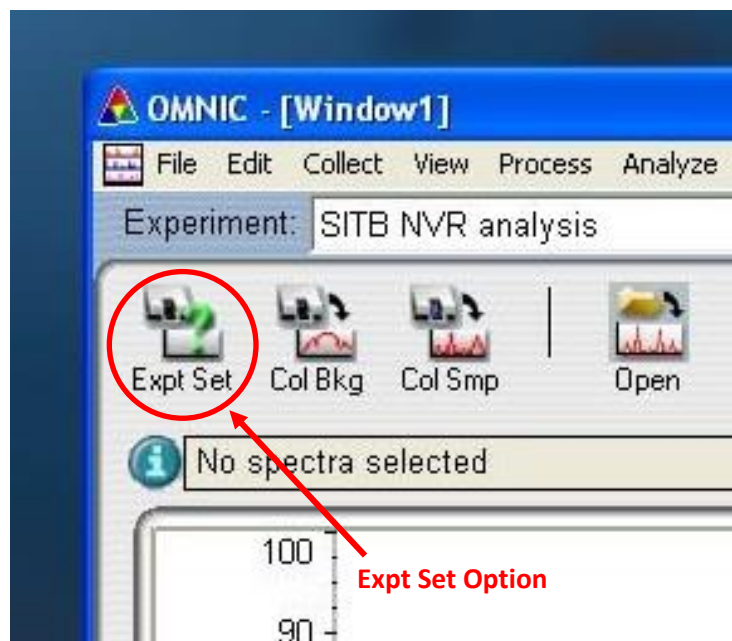


Figure A39. Startup screen for OMNIC.

2. Settings under “Collection” Tab (Figure A40):

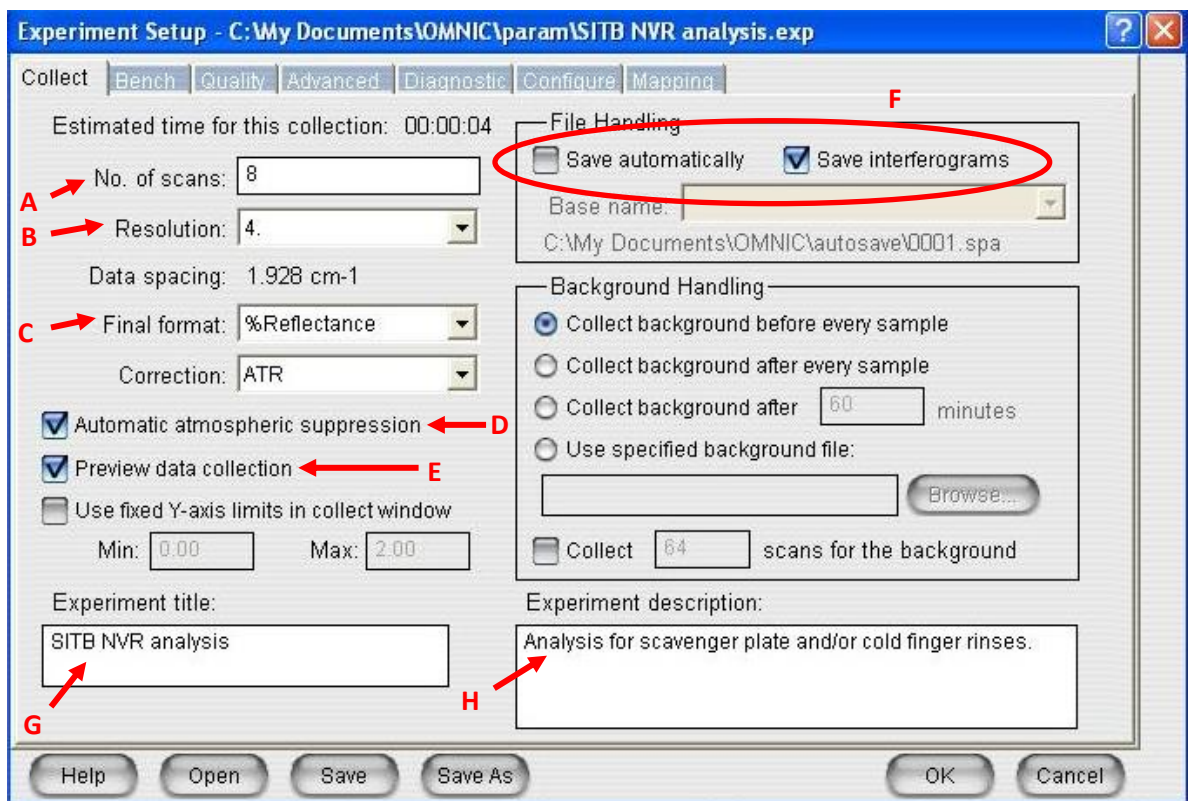


Figure A40. Settings under the Collect Tab in Experimental Setup.

- A. The Number of scans needs to be at least 8.
- B. Resolution is good at 4.
- C. Final format is %Reflectance (ATR uses reflectance when collecting).
- D. Automatic Atmosphere Suppression will take the environment into account during collection (H2O and CO2).
- E. The Preview Data Collection allows for a preview before collection begins.
- F. User does not want to save automatically, but wants to save interferograms.
- G. Assign an Experiment title. This title is what will appear in the drop down menu in the top left of the main screen.
- H. Give an Experiment description.

- Advanced Tab match setting for “SITB NVR Analysis” method (Figure A41).

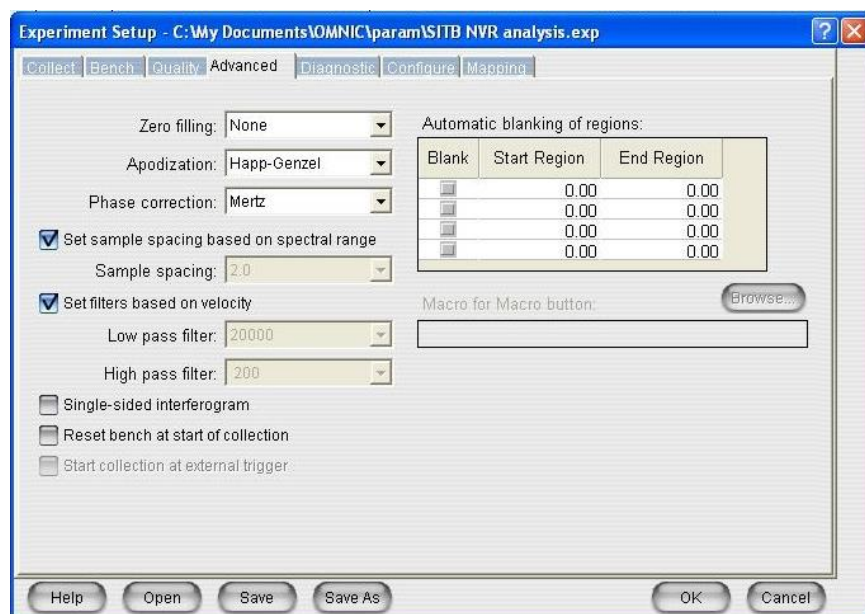


Figure A41. Advanced setting tab options.

- “Bench” tab setup:

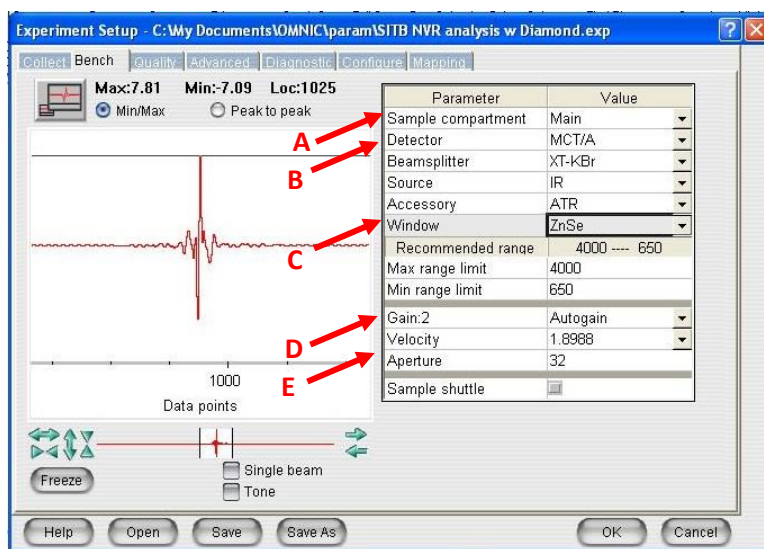


Figure A42. Settings for the Bench tab in Experimental Setup.

- A. For the FTIR Spectrometer set to “MAIN”, the microscope is “SCOPE”.
- B. The detector should be MCT/A.
- C. The Window can be changed between “Diamond” and “ZnSe” depending on which crystal is being used.
- D. Auto Gain allows for the readings to stay within a range. If it is not selected, there could be a peak that exceeds 100%.
- E. Velocity and Aperture are set to company recommended settings.

5. “Diagnostic” Tab (Figure A43):

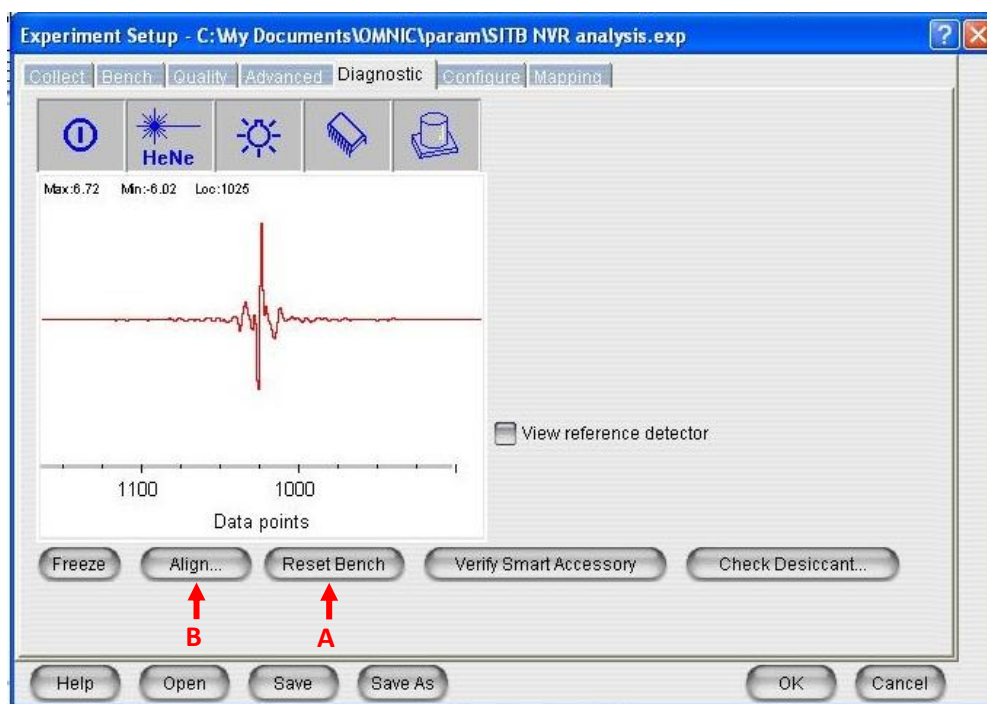


Figure A43. Setting screen for Diagnostic screen.

- a. This can also be opened before filling the FTIR Spectrometer with LN2. The interferogram will increase in intensity as the detector is chilled.
 - b. The procedures only need to be done if the spectrometer has been powered off:
 - A. Reset Bench, if interferogram is not displayed.
 - B. Align.
6. Selecting save will add the method to the Experiment list:

Library Search

NOTE:

This is to be done after peak identification.

1. Check the Library Setup (Figure A44):
 - A. Analysis → Library Setup (Figure A44).
 - B. Check for the following libraries in the “Search libraries and groups” window (right side of the screen):
 - i. Aldrich Condensed Phase Sample Library.
 - ii. Georgia State Crime Lab Sample Library.
 - iii. Hummel Polymer Sample Library.
 - iv. Sigma Biological Sample Library.
 - C. Libraries can be added by selecting the library in the left window and using the “Add >>” button to move them to the right.
 - D. Click on the “Search Results” tab across the top (Figure A45).
 - E. Change the number of spectra to be displayed to match the minimum number of compounds in list.

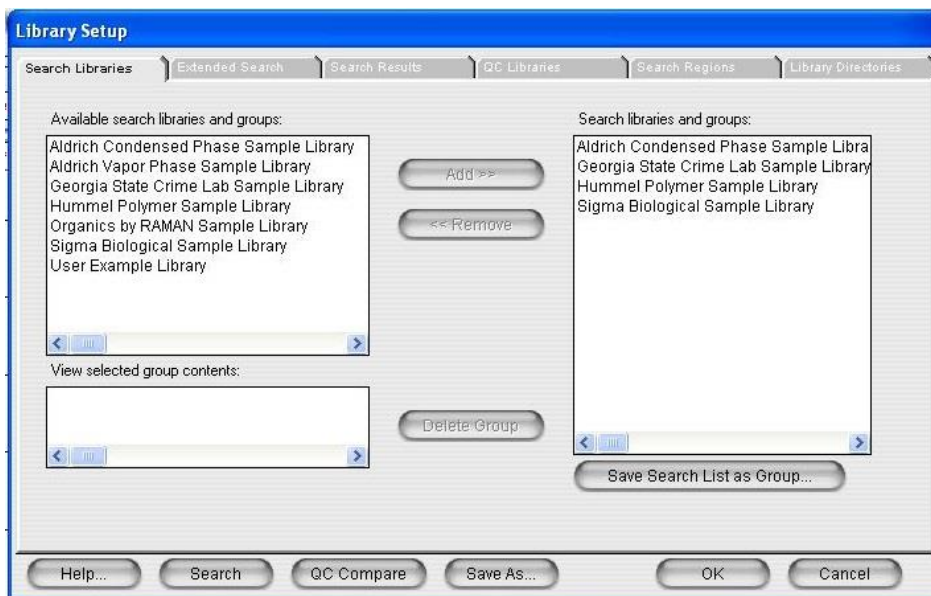


Figure A44. Library Setup with libraries added.

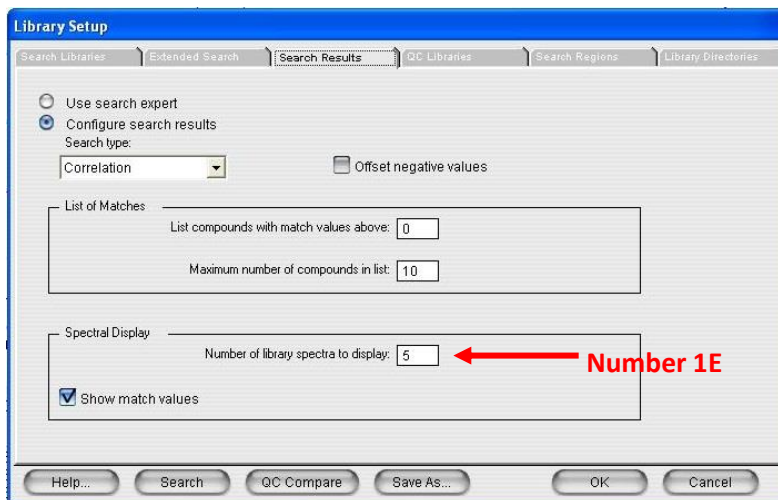


Figure A45. Change the number of search spectra to be displayed.

2. Selecting “Search” will return the compounds that have the closest match to the spectra that is being evaluated (Figure A45).
3. Save the search as a PDF for future reference (See the section about Saving a PDF).
4. After reviewing the results the search result window can be closed by selecting the “Close” button in the bottom right corner (Figure A46).



Figure A46. Library search result page

APPENDIX B: GCMS SOFTWARE OVERVIEW



GCMS Software Overview

Kenneth W. Smith Jr., *LARSS Intern*

April 26, 2013

757.864.4651

Kenneth.W.Smith@nasa.



<http://engineering.larc.nasa.gov/>



Objectives



- **The objective of this presentation is to help assist any persons who will be using the GCMS (Gas chromatography–mass spectrometry).**
- **This presentation will include going through the software and the steps its takes to successfully operate the GCMS.**
- **The set-up and preparation of the samples will be given in another presentation by Patricia Davis and Keith Gordon.**
- **A special thanks to Matt Shacka from Goddard Space Flight Center for his expertise and training.**
- **And a big thank you to Denisse Aranda for making all of this possible.**



Step 1



- From Desktop "Open GCMS Real Time Analysis.





Step 2



- Press OK (you will then hear a beep).
- Press OK again.
- NOTE: Will now see the previous batch appear.

GCMS Real Time Analysis (Admin) [Batch Table - SAGEIII Bx15 Scav QCM1_2.qgb]

File Edit View Instrument Batch Tools Window Help

Folder: C:\GCMSsolution\data\NVRTesting0413

	Vial#	Sample Name	Sample ID	Sample Type	Analysis Type	Method File	Date
1	1	preblank1	preblank1	Q:Unknown		NVRHT1.qgm	04241
2	1	preblank2	preblank2	Q:Unknown		NVRHT1.qgm	04241
3	2	Reference in Chlor	Reference in C	Q:Unknown		NVRHT1.qgm	04241
4	1	referenceblank	referenceblank	Q:Unknown		NVRHT1.qgm	04241
5	3	QCM#1,#2	QCM#1,#2	Q:Unknown		NVRHT1.qgm	04241
6	4	Scavenger Plate	Scavenger Plat	Q:Unknown		NVRHT1.qgm	04241



Process of

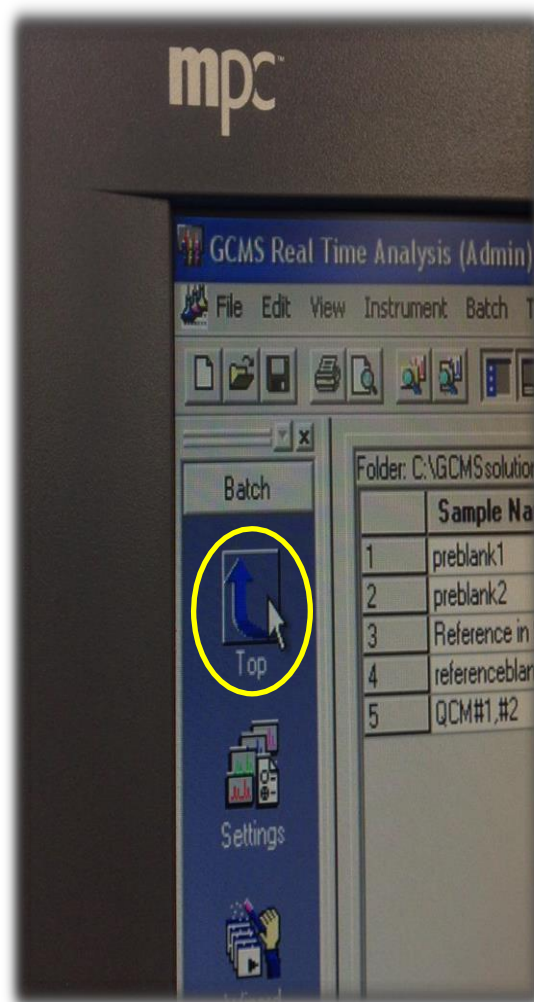
TUNING GCMS



Tuning



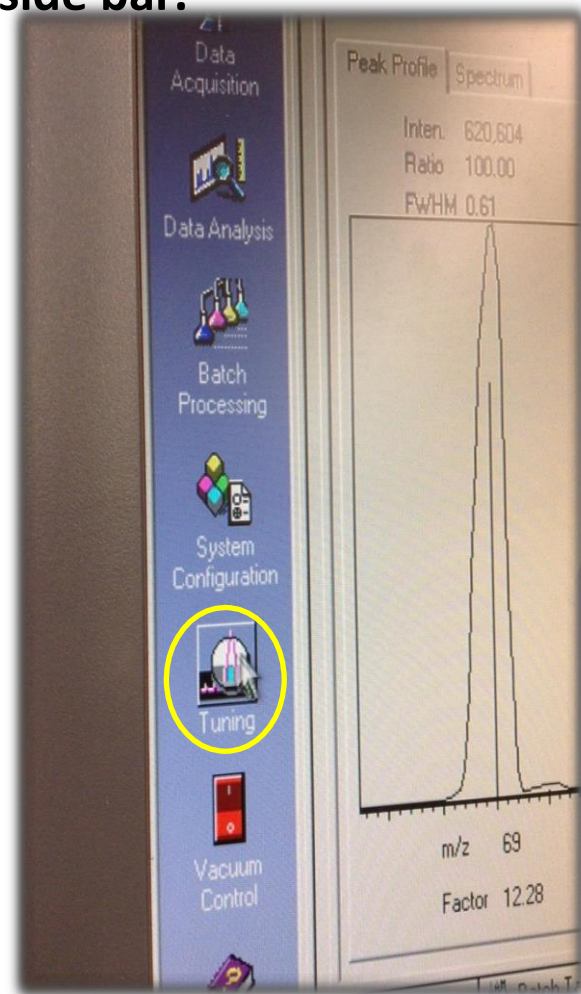
- Before getting started, one must tune the instrument.
- To get started tuning, Click “Top” on the side bar as shown.





Tuning

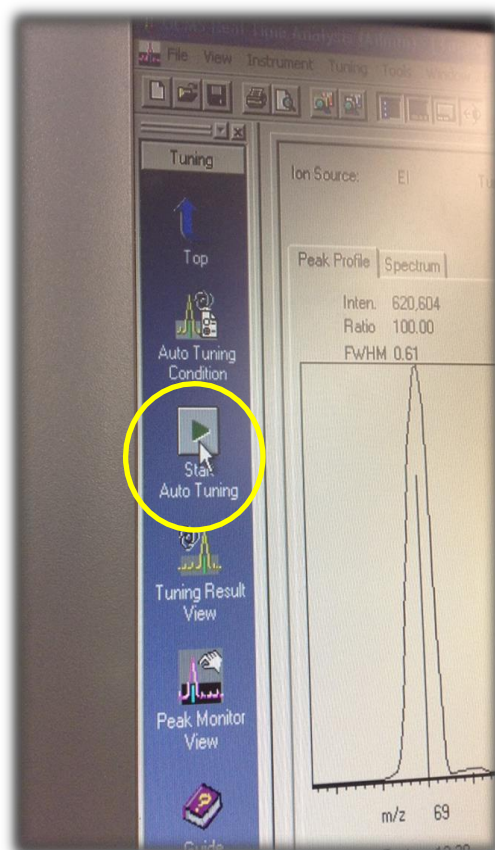
- Click “Tuning” on the side bar.





Tuning

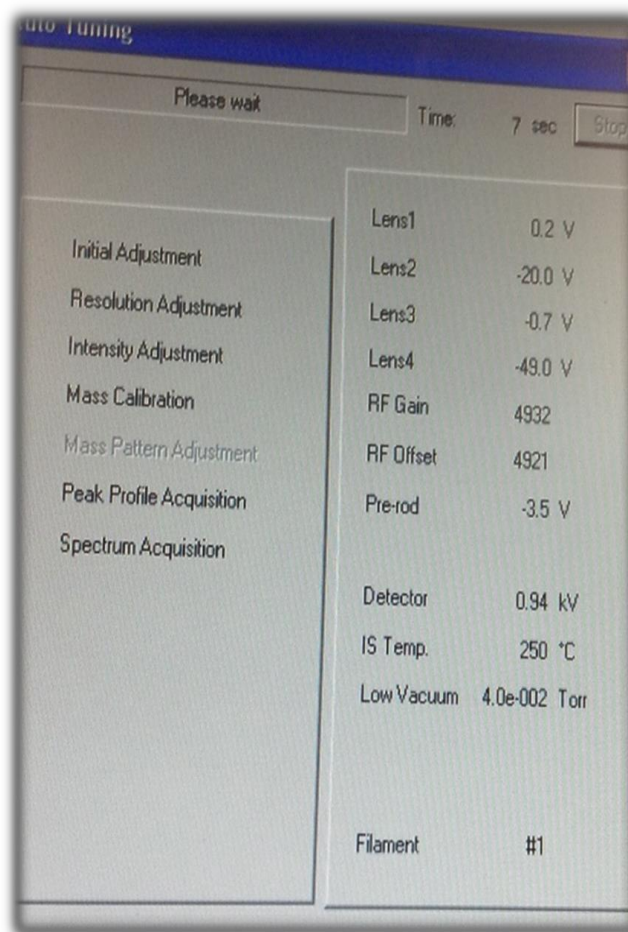
- Click “Start Tuning” on the sidebar on left side of screen.





Tuning

- The following window will now appear.

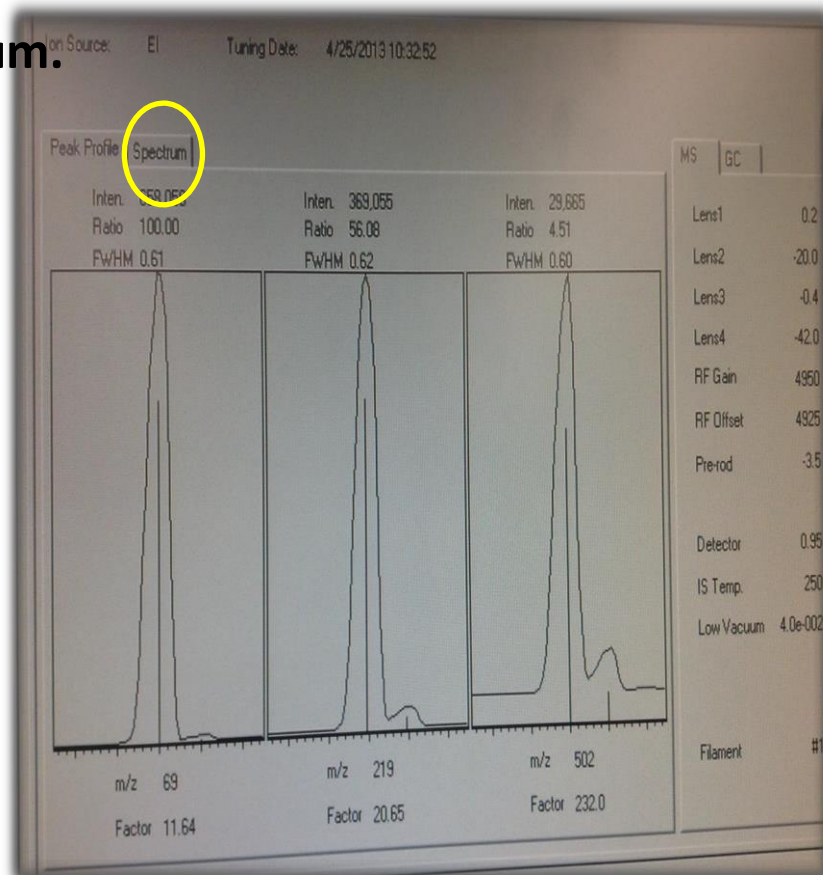




Tuning



- When complete, the following window will appear with two Tabs. Peak Profile and Spectrum.
- Select Spectrum.



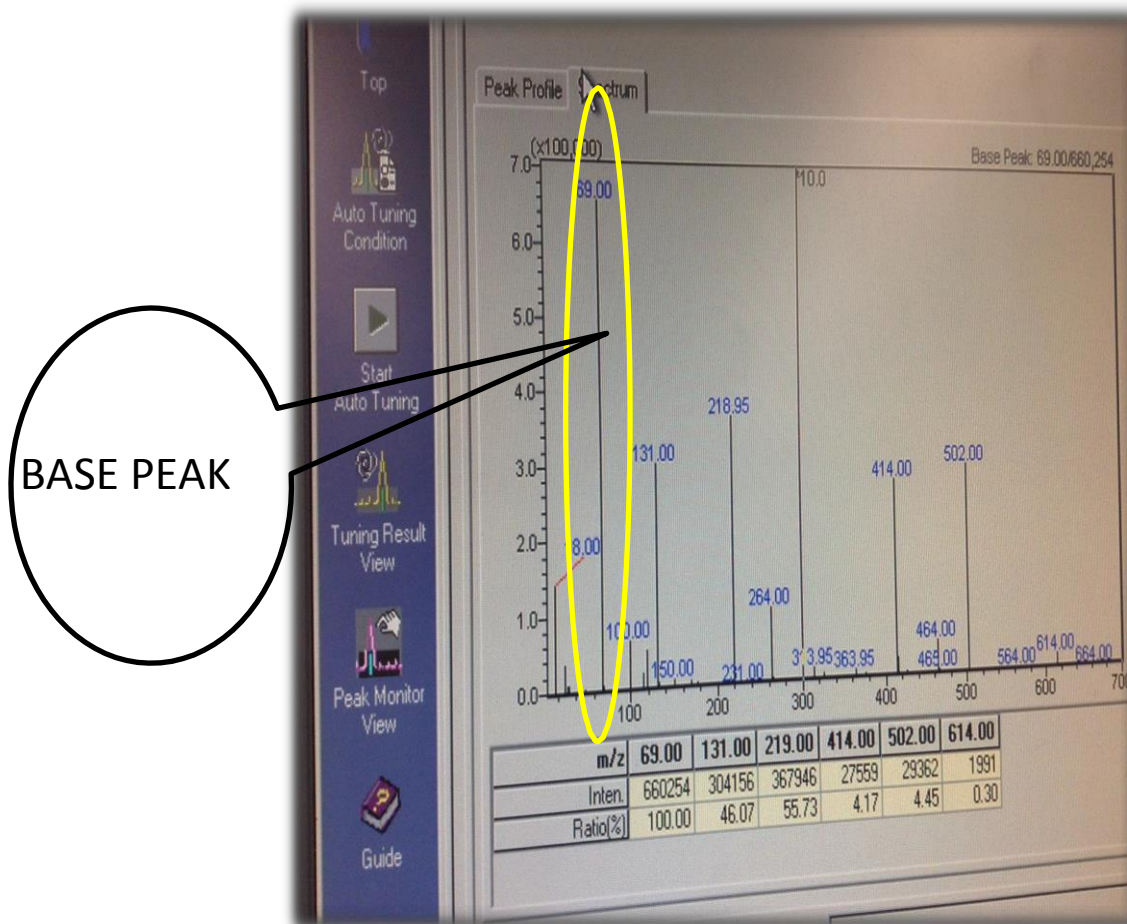


Tuning



➤ If all is well one will see a similar spectrum:

- Make sure 69 is base peak (Biggest).
- 18 and 28 should be considerably smaller.





Tuning

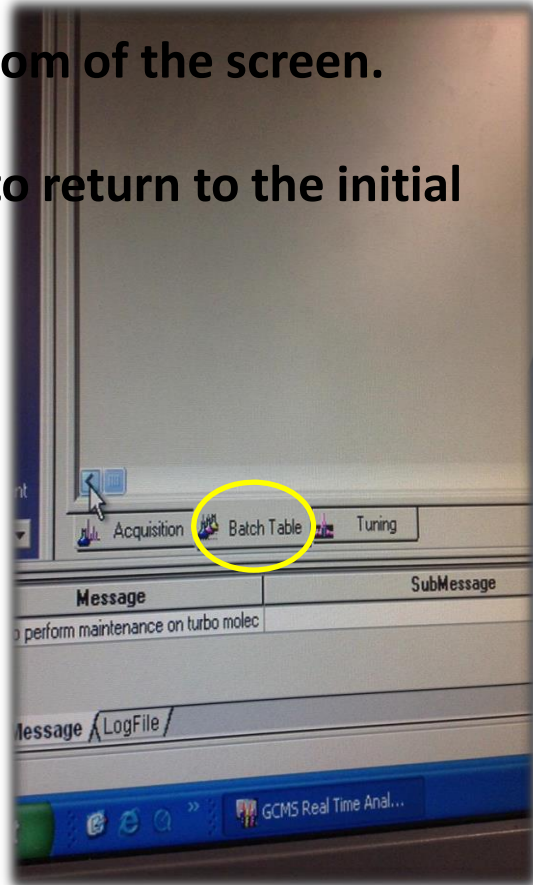


- Please print the Tuning files.
- Write the date on them, then file them away.
- Make sure to **SAVE** the Tuning file; it will be used later!!!
- File→ Save as→ use the following format:
- EI_DATE Example→ EI_042413.qgr:
 - 04 (April) 24 (day) and 13 (2013)



Step 3

- After you save your tuning file, you will see the following tabs at the bottom of the screen.
- Now click “Batch Table” to return to the initial screen.





Understanding the

BATCH TABLE



Overview Look



Analysis (Admin) [Batch Table - SAGEIII 8x15 Scav QCM1_2.qgb]

Instrument Batch Tools Window Help

Folder: C:\GCMSSolution\Data\NVRTesting0413

	Vial#	Sample Name	Sample ID	Sample Type	Analysis Type	Method File	Date
1	1	preblank1	preblank1	Q:Unknown		NVRHT1.qgm	042413
2	1	preblank2	preblank2	Q:Unknown		NVRHT1.qgm	042413
3	2	Reference in Chlor	Reference in C	Q:Unknown		NVRHT1.qgm	042413
4	1	referenceblank	referenceblank	Q:Unknown		NVRHT1.qgm	042413
5	3	QCM#1,#2	QCM#1,#2	Q:Unknown		NVRHT1.qgm	M8x1
6	4	Scavenger Plate	Scavenger Plat	Q:Unknown		NVRHT1.qgm	ESx1

Analysis (Admin) [Batch Table - SAGEIII 8x15 Scav QCM1_2.qgb]

Edit View Instrument Batch Tools Window Help

Folder: C:\GCMSSolution\Data\NVRTesting0413

	Data File	Level#	Inj. Volume	ISTD Aml.	Report Output	Report File	Tuning File
1	042413.qgd	1	1	(Level1 Con	<input type="checkbox"/> Print	ral_report1.qgr	EI_042413.qgt
2	042413.qgd	1	1	(Level1 Con	<input type="checkbox"/> Print	ral_report1.qgr	EI_042413.qgt
3	042413.qgd	1	1	(Level1 Con	<input type="checkbox"/> Print	ral_report1.qgr	EI_042413.qgt
4	042413.qgd	1	1	(Level1 Con	<input type="checkbox"/> Print	ral_report1.qgr	EI_042413.qgt
5	M8x15.qgd	1	1	(Level1 Con	<input type="checkbox"/> Print	ral_report1.qgr	EI_042413.qgt
6	E8x15.qgd	1	1	(Level1 Con	<input type="checkbox"/> Print	ral_report1.qgr	EI_042413.qgt



Overview



- **Vial#** - is the Vial the GCMS will call upon for the sample.
- **Sample Name** and **Sample ID** are just ID Tags for ease of use.
- **Sample Type** - Please keep as 0:Unknown.
- **Analysis Type** - Keep Blank.
 - IF ITQT Selected, go in the dropdown and unselect IT and QT.
- **Method file** - Choose your method:
 - NVRHT1.qgm (use when sample weight is above .34 mg)
 - NVRHT1t.qgm (use when sample is under .34 mg) “more sensitive readings”
 - Bakeout.qgm (for short bakeouts)
 - Bakeoutlong.qgm (for long more thorough bakeouts)
- **Data File**
 - Make a data file that best fits, Template: put date, project name, and what the contaminants came off of.
(like 0424_SAGEIII_8x15TVACScavengerPlate.qgd).



Overview



- **Level # and Inj. Volume - Keep both at 1.**
- **ISTD Amt. - Keep at Level 1 Con.**
- **Report Output - Keep unchecked.**
- **Report File - Change when new report is needed.**
- **Tuning File - This is where you open up the file that was saved when tuning.**
- **Data Description - Keep blank.**



Step 4



- Go to File → Save batch file as → Who its for_ what's it from_date.

- NOW HIT START!



- This process will last anywhere from 90 minutes → Multiple hours:
 - Depends on how many samples!
- Readings will not start until 3 minutes into the process so don't be alarmed when you see nothing at the beginning.



General Post-Run

ANALYSIS



Step 1



- Open “GCMS Postrun Analysis” from desktop.

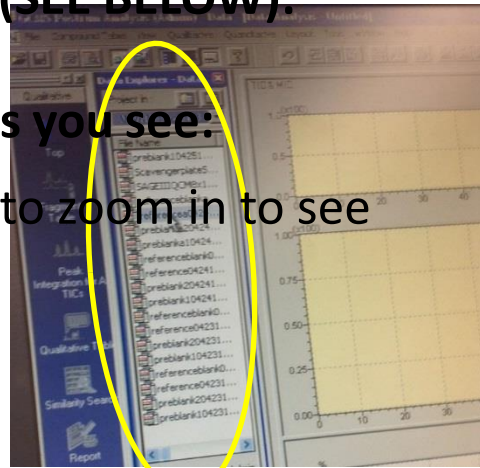




Step 2



- Drop down bar File→Open→ Choose sample need to open from the drop down bar.
- OR use Data Explorer and Navigate and find the sample wanting to analyze (SEE BELOW):
- Count the amount of peaks you see:
 - One could click then drag to zoom in to see peaks better.



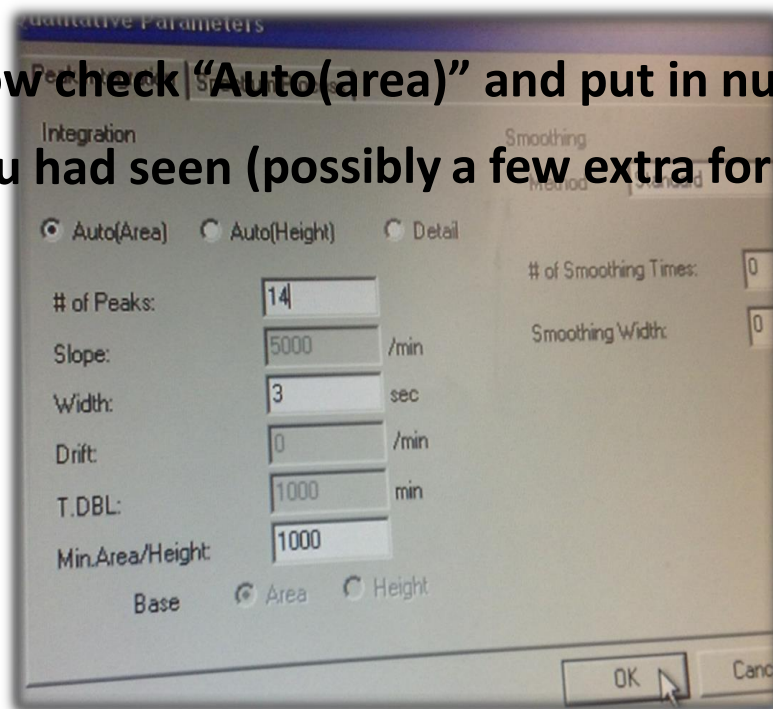


Step 3



- From dropdown, go to qualitative → Peak integrate → TIC...

- Now check “Auto(area)” and put in number of peaks you had seen (possibly a few extra for better analysis).





Step 4



➤ **Please go back and see if the peaks you want are selected:**

- If there is an oversaturation and the machine shuts off. This causes lack of recognition of a significant peak.

➤ **Troubleshoot:**

- If there was an oversaturation 3 times in one run, the run will stop! This means after a point all one will see is a flat line. Please look below, one will see this line after each saturation, but only for a short period unless it is the third saturation point in that run.
- When this happens the sample will now have to be diluted with chloroform. See set-up procedure for dilution instructions.



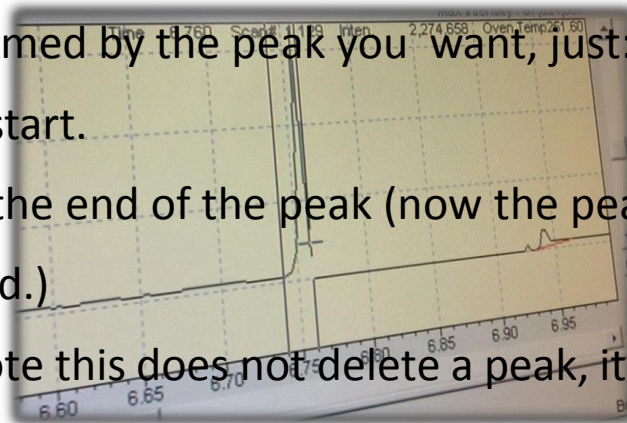
Step 5



➤ If this saturated peak, or any peak you want included, is not selected, one may add peaks manually.

➤ Go to Qualitative → Manual Peaks → TIC

- After zoomed by the peak you want, just:
 - click to start.
 - drag to the end of the peak (now the peak is manually Identified.)
 - Note this does not delete a peak, it is just an addition of a peak.





Step 6



- **After identification a qualitative table must be brought up for identification of peaks.**
- **Go to the dropdown menu Qualitative→ Show Qualitative Table.**
- **A table will now appear with area percentage etc.**



Step 7



- To identify, click the name box for the corresponding peak.
- The peak will appear on the graph and give you the spectrum.



Step 8



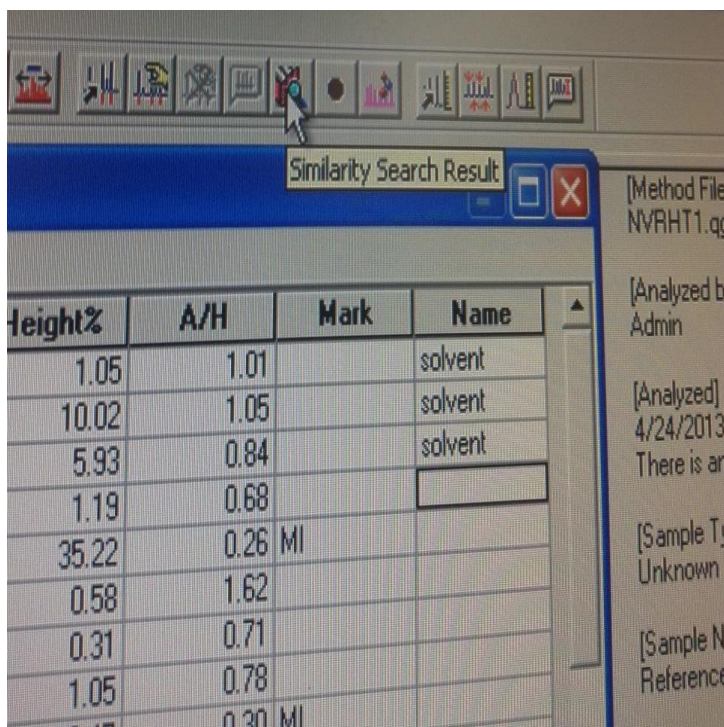
- **Find the base Peak, AKA biggest, as one did in the tuning section.**
- **Look up the base peak in the Goddard reference handout (it is listed in ascending order of base peaks):**
 - Ex., there is a 117 base peak!
 - Flip to the page where 117 is the first number (there is only one)
 - Since there is only one, one can infer that it is “Solvent”
 - HOWEVER if there are multiple, look at other peaks and try to find them on your Spectrum. Please have 2-3 matches in spectrum before considering the material categorization.
 - When found, put name, such as “Solvent” in the name table... this is seen on the next slide.



Step 9



- If an unidentifiable material. Use the similarity search as shown below.
- This will give you a list of possible materials. You can compare spectrums and see what matches.



Height%	A/H	Mark	Name
1.05	1.01		solvent
10.02	1.05		solvent
5.93	0.84		solvent
1.19	0.68		
35.22	0.26	MI	
0.58	1.62		
0.31	0.71		
1.05	0.78		
0.17	0.30	MI	

[Method File
NVRHT1.qg
[Analyzed by
Admin
[Analyzed]
4/24/2013
There is an
[Sample Type
Unknown
[Sample Name
Reference



Rule of Thumb



- By rule of thumb if the total amount of area is less than 1% one may delete the peak categorization.
- **HOWEVER** if it is a silicone, one must keep this because all silicone percentages will be added at the end.



Identifiable Peaks



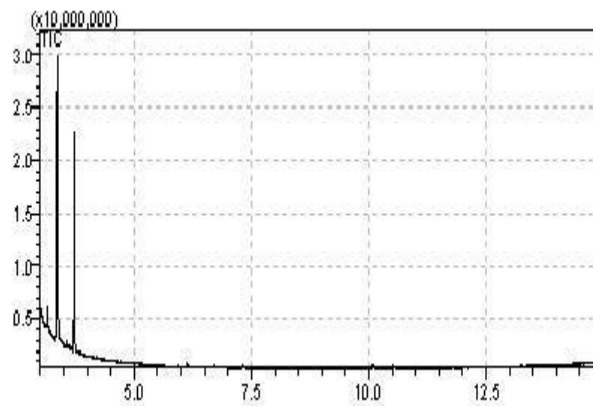
- 117- Will be Solvent (every run will have these thanks to chloroform).
- 73, 147, 221, 355- SILICONE' S.
- 149-Phthalate:
 - For Phthalates base peak is 149, most defining factor for the type of phthalate are the numbers bigger than 149.



Examples

➤ Chloroform Blank:

- Notice minimal peaks.
- Peaks refer to solvent.

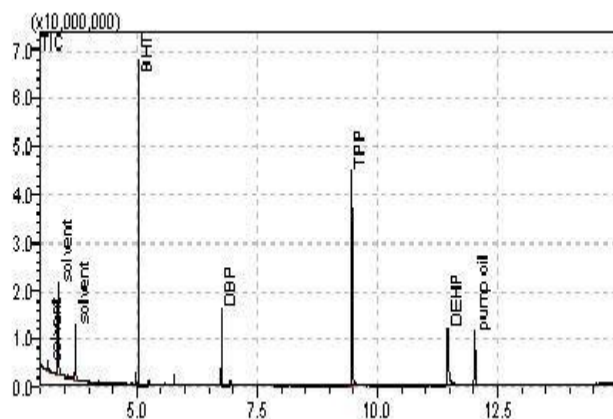




Examples

➤ Reference in Chloroform (made by Goddard):

- Notice solvent peaks at beginning.
- Other easily identifiable peaks.

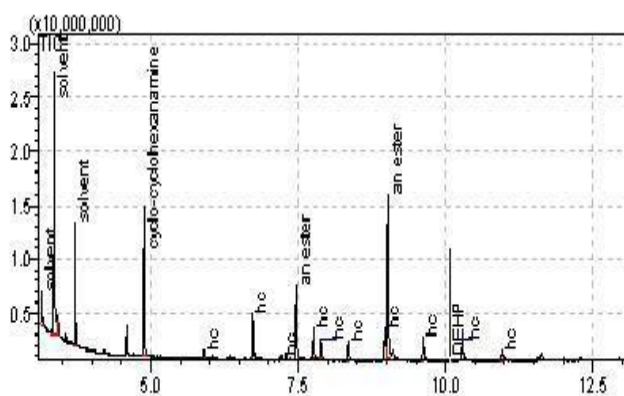




Examples



➤ SAGE III ISS QCM:

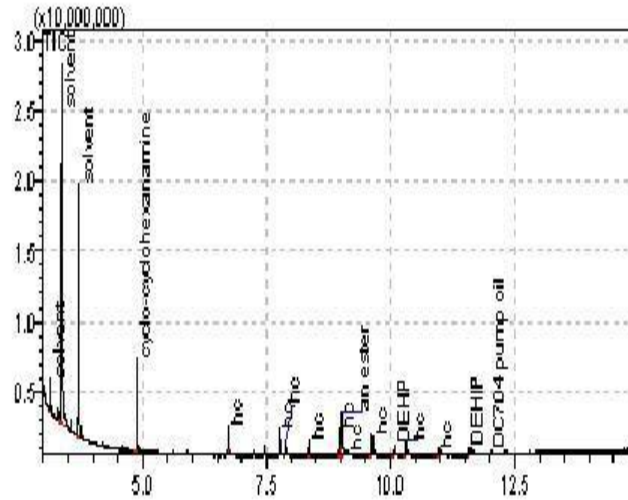




Examples



➤ SAGE III SCAVENGER PLATE:

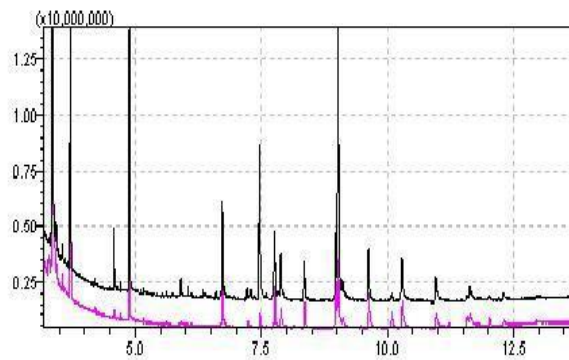




Examples



- **Comparison of SAGE III QCM data and SCAVENGER PLATE data:**





Post Analysis

WRITE-UP



Reports



- **Follow these steps after identification.**
- **Edit→ Select al.l.**
- **Edit Register to spectrum process table.**
- **Then Close Out.**
- **Go to report in left hand side task bar.**
- **Go to data explorer drop down.**
- **Go to reports.**
- **General reports, drag and then drop in.**
- **Print.**



<http://engineering.larc.nasa.gov/>

For more information contact the following person(s):

Keith Gordon

Patricia Davis

Kenneth Smith

For expert help on analysis etc.

Matt Shacka

Contamination Lead Engineer

Denisse Aranda

REPORT DOCUMENTATION PAGE

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					5c. PROGRAM ELEMENT NUMBER	
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14. ABSTRACT Non-volatile residue (NVR), sometimes referred to as molecular contamination is the term used for the total composition of the inorganic and high boiling point organic components in particulates and molecular films deposited on critical surfaces surrounding space structures, with the particulate and NVR contamination originating primarily from pre-launch operations. The "nonvolatile" suggestion from the terminology NVR implies that the collected residue will not experience much loss under ambient conditions. NVR has been shown to have a dramatic impact on the ability to perform optical measurements from platforms based in Space. Such contaminants can be detected early by the controlled application of various detection techniques and contamination analyses. Contamination analyses are the techniques used to determine if materials, components, and subsystems can be expected to meet the performance requirements of a system. Of particular concern is the quantity of NVR contaminants that might be deposited on critical payload surfaces from these sources.						
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